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PROCEEDINGS

PART II

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METHODS OF TEST IN RELATION TO FLOW IN STEELS AT VARIOUS TEMPERATURES[1]

By H. J. FRENCH[2]

SYNOPSIS

This paper relates to flow in a low-carbon structural steel, a high-chromium "stain-resisting" steel and a low-tungsten high-vanadium high-speed steel, when subjected to fixed tension loads at temperatures between 70 and 1350° F. (20 and 730° C.). The loads and temperatures were maintained for varying periods up to five months depending upon test conditions and the results are correlated with factors which may be determined in the customary short-time tension tests.

It was found that the proportional limit, determined in the short-time tension test by an accurate extensometer which is briefly described, approximated the loads permitting long life with freedom from deformation in the flow tests at corresponding temperatures. Within the temperature range in which strain hardening is observed this is lower than the "limiting creep stress" referred to by other investigators; at higher temperatures it is equivalent to the latter.

The report records the adaptation of the Tuckerman-Martens extensometer to high-temperature tension tests.

INTRODUCTION

The purpose of this paper is to present new data concerning so-called "creep" or flow in ferrous metals when subjected for long periods to fixed loads in tension at approximately constant temperatures and to discuss load-carrying ability in relation to factors which may be determined in the customary tension tests.

The need for additional information in this field was clearly demonstrated at the 1924 Symposium on Effect of Temperature on the Properties of Metals held jointly by the American Society of Mechanical Engineers and the American Society for Testing Materials **(1)**,[3] and by subsequent discussions at the 1925 annual meeting of the latter society(2). The tests to be described are similar to those which are now being carried out in a much more extended program under the auspices of the Joint Research Committee, of the two societies, on Effect of Temperature on the Properties of Metals, and were begun about 2½ years ago. The author's tests are restricted to three steels which, however, differ widely in chemical composition as shown in Table I.

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[1] Published by permission of the Director of the U. S. Bureau of Standards, Department of Commerce, Washington, D. C.

[2] Metallurgist, U. S. Bureau of Standards, Department of Commerce, Washington, D. C.

[3] The boldface numbers in parentheses refer to the papers given in the list of references appended hereto.

PREVIOUS STUDIES OF "CREEP" IN METALS

The fact that metals flow or "creep" when subjected to sufficiently high stresses is not a recent discovery; likewise it has long been known that the "time factor" is of much greater importance in steels at "red heat" than at atmospheric or slightly elevated temperatures. Not until quite recently, however, has much attention been paid to so-called "creep," "flow," "long-time" or "sustained-loading" tests and then largely because of condemnation by certain investigators of the older and more generally used tension tests, arising from the fact that continuous "creep" occurs at stresses below the tensile strength values at corresponding high temperatures.

TABLE I.—CHEMICAL COMPOSITIONS OF THE STEELS TESTED.

ELEMENT	OPEN-HEARTH BOILER PLATE[a]	HIGH-CHROMIUM STEEL WITH COPPER[b]	HIGH-SPEED STEEL[c]
Carbon, per cent........	0.24	0.28	0.77
Manganese, per cent.....	0.37	0.38	0.24
Phosphorus, per cent....	0.021	0.026	0.031
Sulfur, per cent.........	0.028	0.013	0.035
Silicon, per cent.........	0.01	0.17	0.42
Chromium, per cent.....	....	20.5	3.9
Tungsten, per cent......	....	....	13.6
Vanadium, per cent.....	....	....	1.9
Copper, per cent........	....	0.98	....
Nickel, per cent.........	....	not detected	0.14

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[a] Tested in the hot-rolled condition; the plate was 1 in. thick.

[b] Water-quenched from 1830° F. (1000° C.) and then tempered at 1200° F. (650° C.) before test.

[c] Tested in the condition "as received" from the manufacturer; probably annealed subsequent to hot working.

The change from the customary tension tests to "creep" tests has not wholly clarified the situation; apart from variations in the manner in which such tests have been carried out, the stresses below which continuous "creep" is not found have frequently been greatly in excess of the maximum stresses which engineers dare to use in service. An example of this is shown in Fig. 1. Likewise, metallurgists are not all convinced that ordinary tension tests, when properly carried out, will not give useful information concerning the load-carrying ability of metals at high temperatures for the type of service considered.

However, since the older test methods have been criticized it seems necessary first to study flow in metals under loads for long periods and subsequently to correlate results with factors which may be secured in the less expensive and less laborious tension or other quick tests.

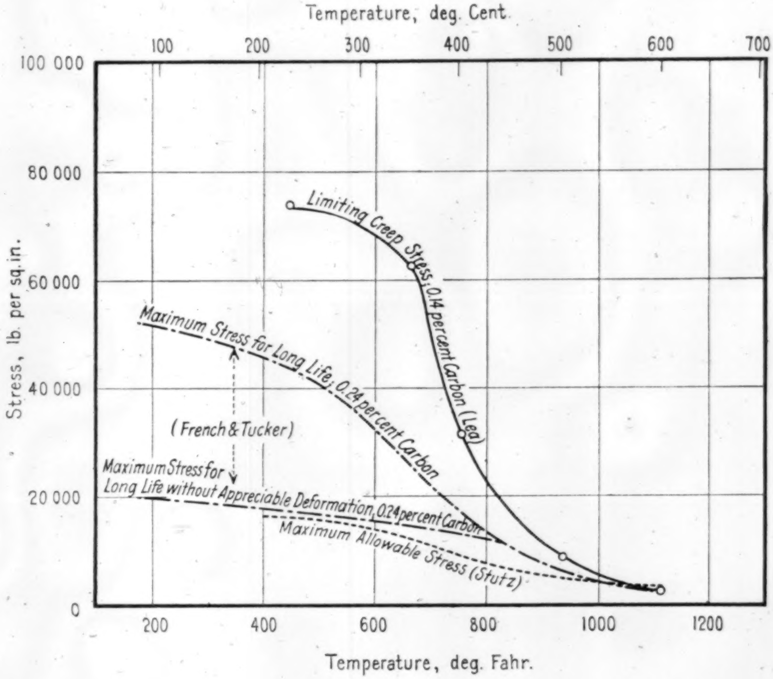

FIG. 1.—Comparison of Some "Design Load Values" Used Industrially with "Limiting Creep Stress" and Results Obtained in the Author's Flow Tests for Low-Carbon Steels at Different Temperatures.

The maximum allowable stress values given by Stutz are stated to have been used successfully for many years in the design of structures for high-temperature service. They refer to the same type of steel as that tested by the author. Note the relatively high values of the "limiting creep stress" found for lower carbon steel by Lea throughout the major portion of the temperature range considered. For details concerning the origin of these data, see references (6) and (7).

Previous "creep" tests may be grouped under two main headings as follows:

1. Tests in which the applied load is fixed:
   (*a*) at constant temperature;
   (*b*) under changing temperature; and
2. Constant maximum-stress - constant-temperature tests.

Included in the first group are the tests reported by Le Chatelier(3), Cheyenard(4), Dickenson (5), Lea(6), the author(7), Lynch, Mochel and McVetty(2), Tapsell and Bradley(8), and Bailey(9). The procedure

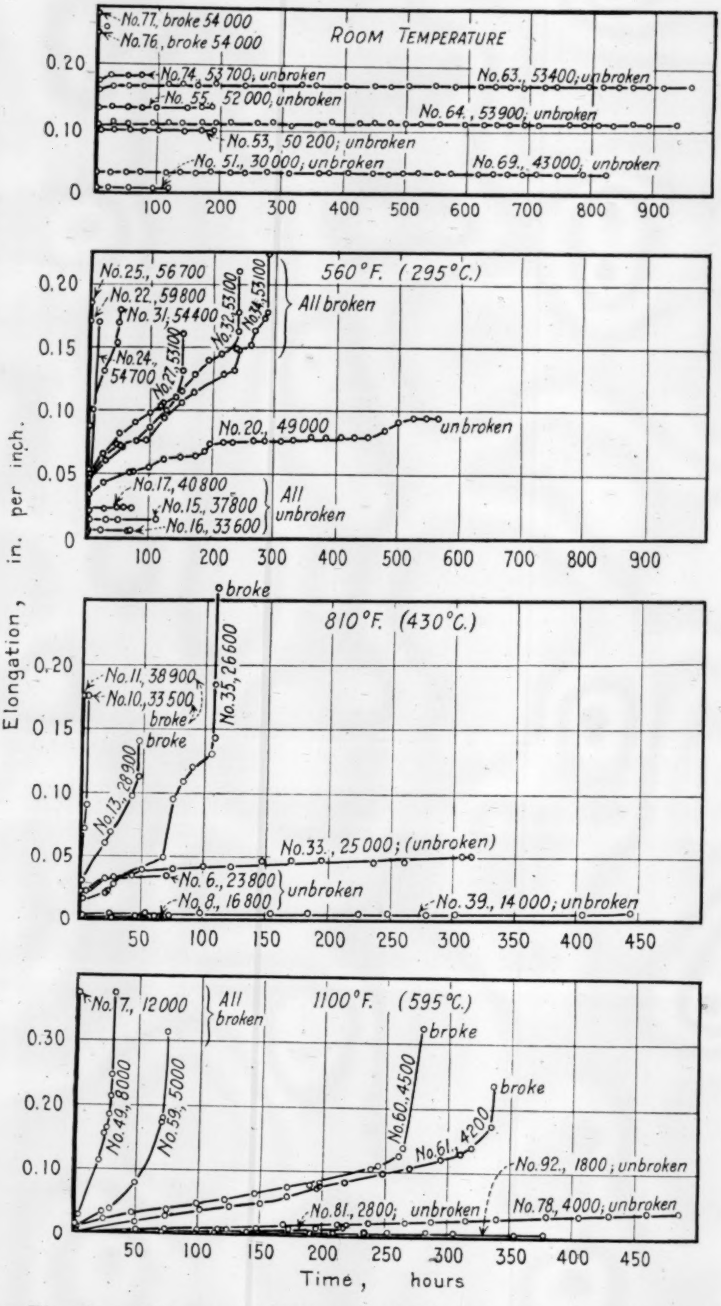

FIG. 2.—Flow in a 0.24-per-cent Carbon Steel Under Sustained Tension Loads at Various Temperatures.

The numerical values given above each of the time-elongation curves refer to the applied load expressed in pounds per square inch of the original cross-section. For the chemical composition of this steel, see Table I.

used by Dickenson consisted in gradually increasing the temperature until appreciable flow was observed and in this way determining the limiting temperatures below which the metal could for long periods sustain a particular load without appreciable deformation. The objection which may be raised to such procedure is that only partially quantitative data are obtained and not the maximum loads which can be sustained at different temperatures. The earlier and more recent investigators mentioned followed the flow under fixed applied loads and approximately constant temperatures.

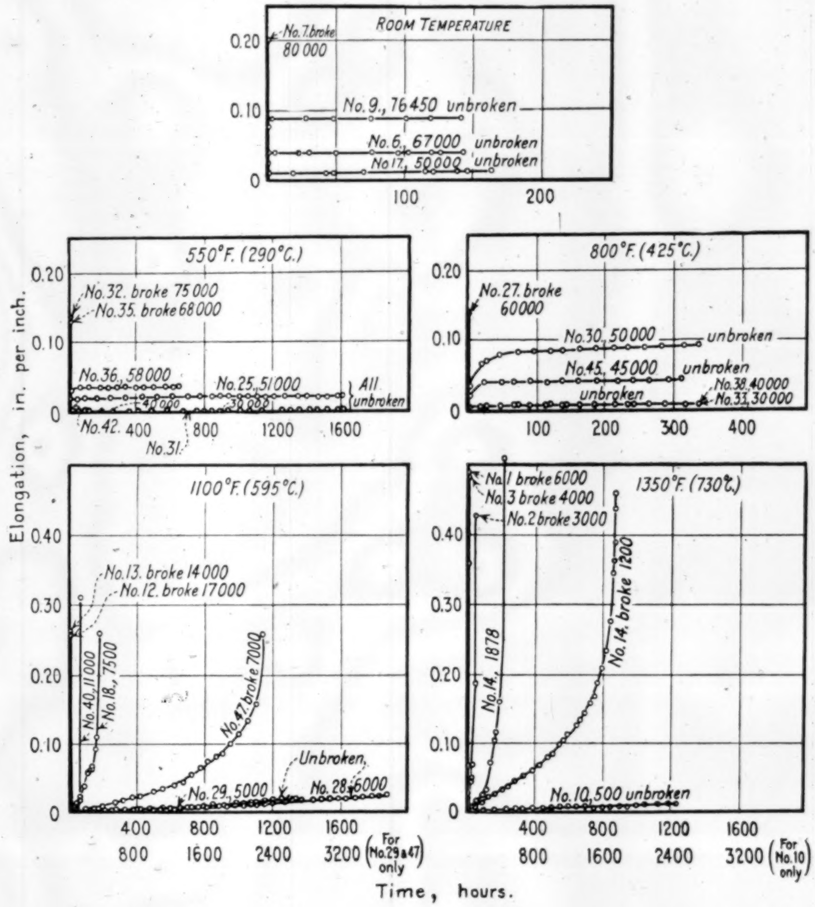

FIG. 3.—Flow in a High-Chromium "Stain-Resisting" Steel Under Sustained Tension Loads at Various Temperatures.

The numerical values given above each of the time-elongation curves refer to the applied load expressed in pounds per square inch of the original cross-section. For the chemical composition of this steel, see Table I.

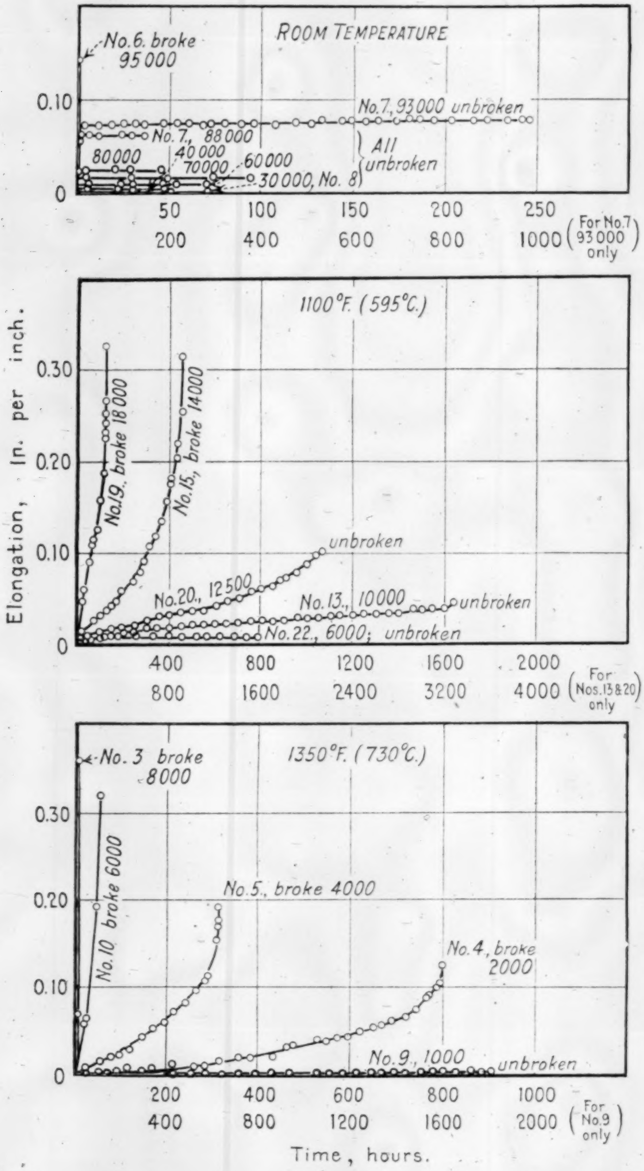

FIG. 4.—Flow in a Low-Tungsten, High-Vanadium High-Speed Tool Steel Under Sustained Tension Loads at Various Temperatures.

The numerical values given above each of the time-elongation curves refer to the applied load expressed in pounds per square inch of the original cross-section. For the chemical composition of this steel, see Table I.

In the second group are the tests quite recently reported by Cournot and Sasagawa(10) in which the maximum stress was kept constant by special equipment designed to reduce the total load as the specimen stretched and decreased in cross-section.

With a few exceptions, including the tests recently reported by the author which have been confirmed by Malcolm(11) in a limited study of cast nickel-chromium steel, previous studies have mainly given the conception that for each material at each temperature there is a definite load above which flow is continuous and below which it is not. This, however, is not a complete picture as will be shown by the tests to be described.

EXPERIMENTAL RESULTS

Character of Flow Under Fixed Applied Loads:

Tests already reported for low-carbon structural steel(7) showed that the total flow resulting in fracture took place in three stages, the magnitude and importance of which varied with the applied load and temperature (see Fig. 2). These three stages of flow were designated (1) an initial flow; (2) a secondary flow, at somewhat lower rate than that observed in the first and third periods; and (3) a final rapid flow just before fracture. Comparison of Figs. 2, 3, and 4 shows that these comments are equally descriptive of the flow in the high-chromium "stain-resisting" steel and the high-speed steel. Likewise, as the fixed applied load is increased the initial flow and the rate of flow in the second period increase and the life of the steel decreases. The final rapid flow begins when the reduction in cross-section accompanying appreciable elongation has raised the unit stress to a definite load at each temperature for each metal.

At atmospheric temperatures there is a small difference between the maximum loads permitting very long life and those producing fracture in a few moments. As the temperature is raised the increase in life with decrease in load becomes more gradual, and in place of an initial flow of appreciable magnitude followed by what appears to be dimensional stability there is either continuous flow or freedom from any appreciable deformation.

Cessation or very marked reduction in flow subsequent to initial deformation at atmospheric and slightly elevated temperatures may be attributed to strain hardening and permits very long life at relatively high stresses. Under the described test conditions, the strain-hardening ability is greatest at atmospheric temperatures but its rate of decrease and final disappearance with increase in temperature vary in the several steels.

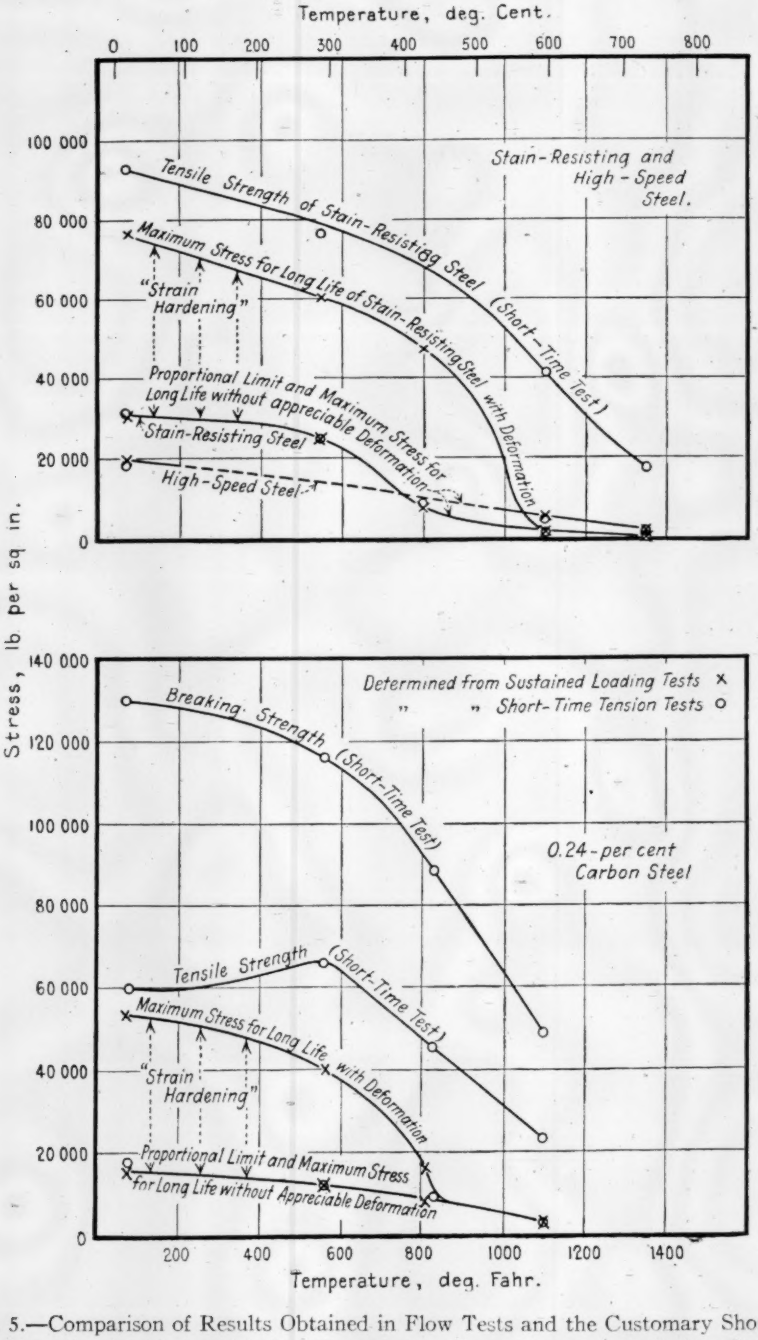

FIG. 5.—Comparison of Results Obtained in Flow Tests and the Customary Short-Time Tension Tests at Various Temperatures for a 0.24-per-cent Carbon Steel, a High-Chromium "Stain-Resisting" Steel and a High-Speed Tool Steel.

For the chemical compositions and preliminary heat treatments of the three steels, see Table I.

These changes are of considerable importance for they show that the principal factor governing maximum allowable stress in a given steel varies with the temperature and the type of service. Throughout the temperature range in which strain hardening is observed, somewhat higher stresses can be sustained when long life is the only requirement than when long life and freedom from deformation are both important, as is most often the case in practical service; above the strain-hardening temperature range long life is only obtained with freedom from appreciable deformation.

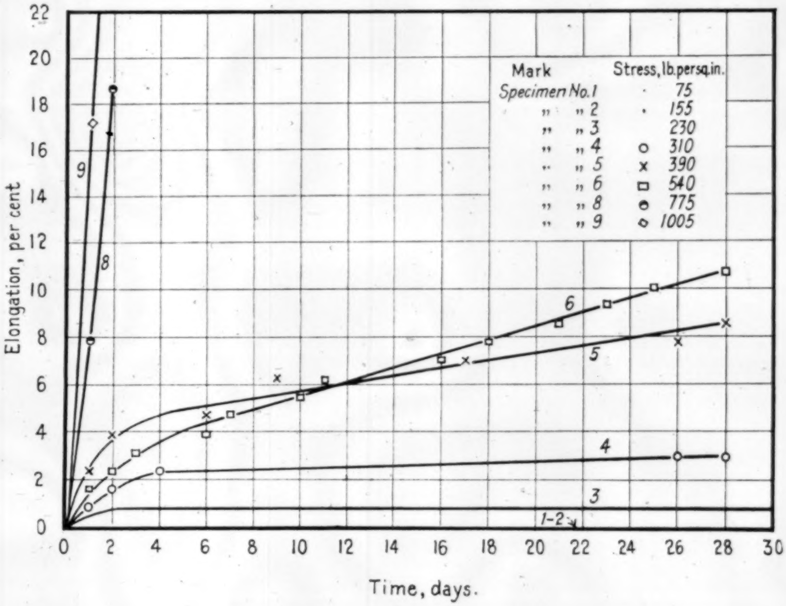

FIG 6.—Flow in Tin Under Sustained Tension Loads at Atmospheric Temperatures as Reported by Freeman and Quick.

For the origin of these data, see reference (12).

A graphical summary of these features for the three steels studied is given in Fig. 5. The loads permitting long life without appreciable deformation were obtained by interpolation or extrapolation from initial-flow - load curves at temperatures within the strain-hardening ranges and from curves representing the relation between the secondary flow rate and the load at all higher temperatures. There is, of course, no sharp dividing line between the several stages of flow nor is the rate wholly uniform in any one stage, so that this procedure is merely an aid in the approximation of the desired values from data shown in Figs. 2, 3 and 4.

It is interesting to note that the time-elongation curves of the three steels at temperatures above the strain-hardening range are quite similar to those for tin and certain lead-tin alloys (solders) at atmospheric temperatures. This is plainly shown by comparison of Figs. 2, 3, and 4 with Fig. 6 reproduced from a recent report on soldered joints by Freeman and Quick(12).

Rapid Approximation of Load-Carrying Ability:

Tests such as those described give a fairly complete picture of flow under sustained loads at different temperatures but require considerable time and are therefore expensive, particularly if an accurate determination of load-carrying ability is desired. It is not desirable, for example, to have to follow for long periods the behavior of metal specimens selected to represent parts to be subjected to high-temperature service. But, aside from inspection problems, there are now many metals used at various temperatures for which no very complete data are available, and to study each of these in the manner described would require more time and concentrated effort than can justly be put upon

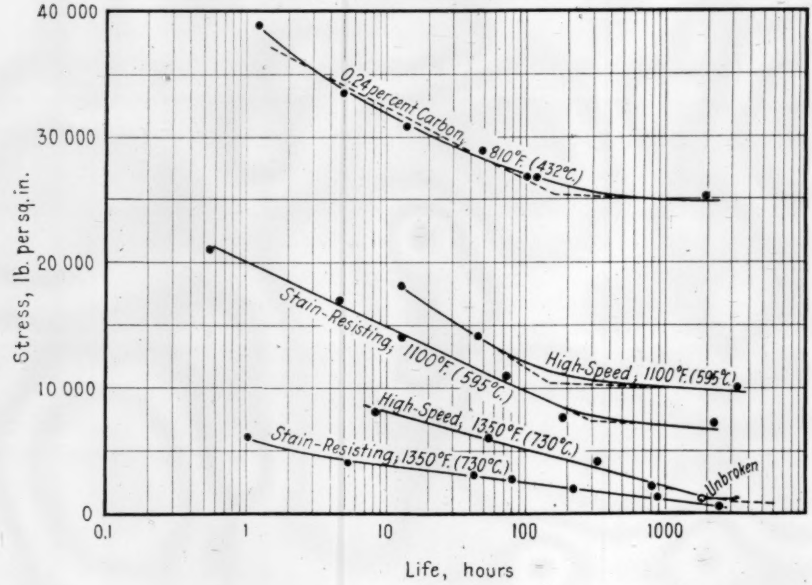

FIG. 7.—Relation Between Applied Load and the Life of a 0.24-per-cent Carbon Steel, a High-Chromium "Stain-Resisting" Steel, and a High-Speed Tool Steel at Various Temperatures.

Note that the life is plotted logarithmically. The dotted lines serve to emphasize a bend or "knee" in the curves. For the chemical compositions of the steels, see Table I.

so-called flow tests which relate to but one phase of a very broad problem. It is therefore important to seek more rapid test methods which will indicate load-carrying ability for the conditions considered.

At least two methods of attack are available: (*a*) the development of a general relation between load, life and deformation by means of which the results from a relatively quick fixed-load test may be translated into terms of long life; or (*b*) a study of the relation between flow tests and factors determinable in the customary tension tests in which fracture is usually obtained in a few moments by progressive increase in stress.

(*a*) *Quick Fixed-Load Tests.*—The relation between applied load and life, when plotted to semi-logarithmic coordinates as shown in Fig. 7, or in other ways, bears a strong resemblance to the stress-cycle relations of fatigue tests at atmospheric temperatures (so-called S-log N diagrams, in which S refers to stress and N the number of repetitions). While there are hardly enough points to establish the exact form of all these curves, taken collectively they indicate a bend or "knee" in load-life curves. Furthermore, this knee appears to be shifted to longer life as the temperature is raised in a given alloy or, in comparison of the three alloys at a given temperature, it is at the longest life in the one which has the lowest load-carrying ability. In other words, the higher the temperature the more tedious is the accurate determination of long-life loads. For example, tests continued for periods up to 2500 hours with the stain-resisting steel at 1350° F. (730° C.) gave no evidence of a "knee" in the load-life curve whereas at 1100° F. (595° C.) a well-defined change in direction is found in the neighborhood of 300 hours life.

Whether or not the load-life curve becomes horizontal to the right of the bend or continues downward at a very slow rate has, of course, not been determined and seems to be relatively unimportant from the standpoint of this discussion; in either case the metal has a real load-carrying ability. The important feature is that the existence of a bend makes difficult the development of a general law covering load-life relations by which relatively quick tests can invariably be translated into long life conditions.

It has already been pointed out that the flow resulting in fracture takes place in three stages and that the final stage is the result of stress concentration brought about by a reduction in cross-section accompanying the previous elongation. Obviously, if the flow rate in the second period is equal to zero this stress concentration will not occur and the specimen should theoretically never break; at least, it should have a very long life. This then appears to offer a quick method of determining loads permitting long life either with or without appreciable deformation. Practically, however, two difficulties are encountered.

Within the strain-hardening temperature range of a metal a very low rate of flow in the second stage may be preceded by an appreciable initial flow. The strain hardening which stops this is not instantaneous and the initial flow may continue for days, thus prolonging the time necessary for the desired measurements. This difficulty is, of course, not encountered above the strain-hardening temperature range, but under such conditions, as well as at lower temperatures, very accurate measurements and control of test conditions are required to determine the load-carrying ability in a short time. For example, in the low-carbon structural steel at 810° F. (430° C.), fracture is not far distant when the total elongation is in the neighborhood of 6 to 10 per cent (see Fig. 2). If it is assumed for the moment that the flow up to this point takes place at a uniform rate, then the rate of flow for a life of 10,000 hours would be about 6 to 10 millionths of an inch per inch per hour. This is, of course, not an accurate illustration but serves

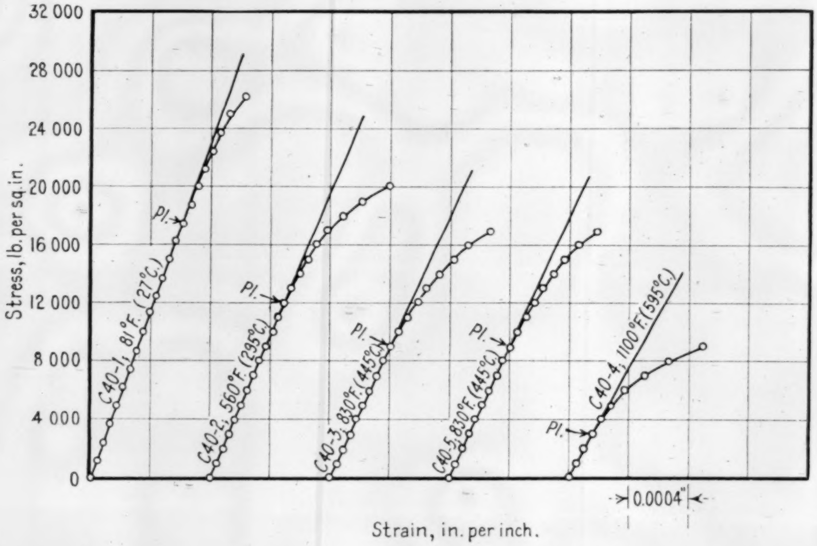

FIG. 8.—Stress-Strain Diagrams for a 0.24-per-cent Carbon Steel at Various Temperatures.

These results were obtained by McVetty of the Westinghouse Electric and Manufacturing Co. with the extensometer described by Wilhelm. For the origin of these data and other details, see references (1) and (7).

P.L. refers to the selected value of the proportional limit. For the chemical composition of the steel, see Table I.

to show the order of magnitude of changes which must be dealt with in tests of short duration.

(*b*) *Comparison of Flow Tests and Tension Tests.*—The previous discussion indicates that it is possible to approximate load-carrying ability in much shorter time than has been taken in the described flow tests, but even under favorable conditions with respect to the temperature and metal tested more time is required than is necessary for a tension test in which fracture is generally produced in a few moments by progressive increase in stress. Therefore, any relation between the results obtained from the two methods of test becomes of considerable importance, even though an approximation.

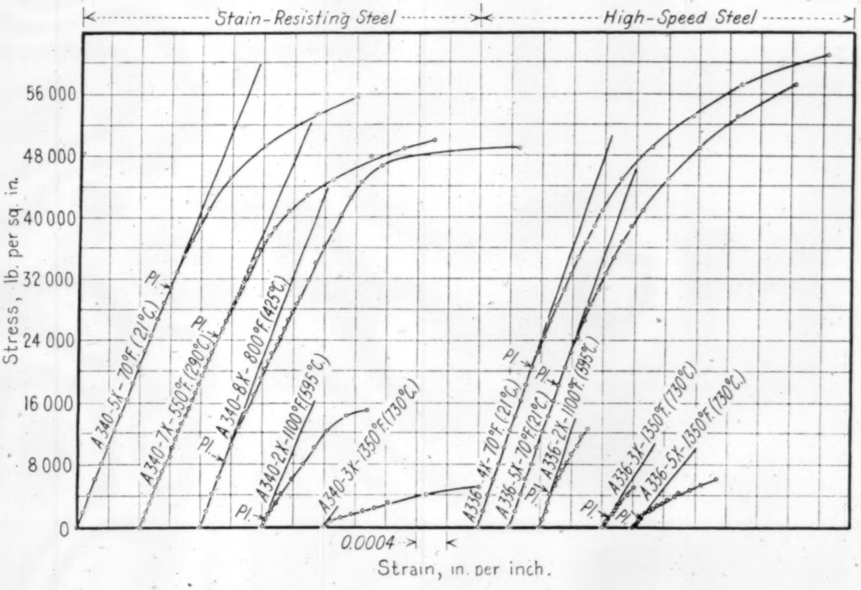

FIG. 9.—Stress-Strain Diagrams for a High-Chromium "Stain-Resisting" Steel and a High-Speed Tool Steel at Various Temperatures.

These results were obtained with the Tuckerman-Martens extensometer shown in Fig. 10.

P.L. refers to the selected value of the proportional limit. For the chemical compositions of the steels, see Table I.

In a previous discussion of results for low-carbon steel (7) and in a report by Lynch, Mochel and McVetty, (2) it was shown that the proportional limit of certain ferrous metals approximated the loads permitting long life at corresponding temperatures without appreciable deformation. Data included in this report give further support to and extend the usefulenss of this relation, as will be evident from comparisons of Fig. 5 with Figs. 8 and 9. It is too early to predict to what extent or with what accuracy this may apply, but it would be reasonable to expect that it would hold quite generally for steels initially in a stable condition for the temperatures considered. Preliminary indications obtained by Lynch, Mochel and McVetty indicate that non-ferrous metals will probably have to be considered in a different light although future work may alter this viewpoint. Bailey(9) is now of the opinion that "at any temperature at which removal of strain hardening occurs, the limiting creep stress—that is, the stress below which there is no creep—would be expected to be the elastic or

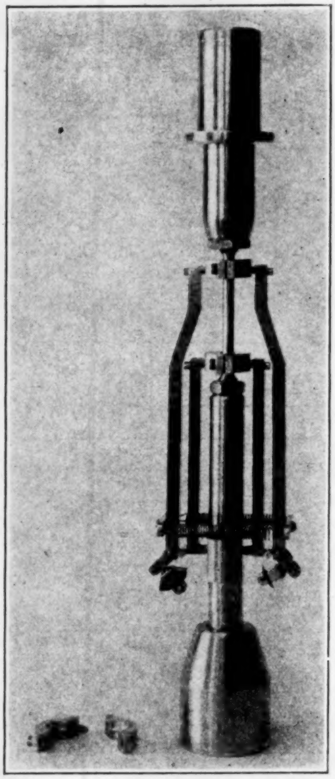

FIG. 10.—The Tuckerman-Martens Extensometer Adapted for High-Temperature Tension Tests and Used in the Tests Described.

A direct reading of strain of 0.000004 in. is obtained with this instrument and the autocollimator used.

The collars and frames are made of a nickel-chromium-iron alloy having good resistance to oxidation in air at high temperatures. For a description of the principles of construction, see reference (**14**).

proportional limit," but few investigators have studied this question experimentally. Since such a relation exists in ferrous metals differing so widely in chemical and physical properties as the three steels used in this investigation it seems important to include, in future studies of "creep," determinations of the stress-strain relations in tension tests.

However, if such a correlation is attempted, it should be recognized that the numerical values of proportional limit are dependent to some extent upon the methods of plotting results and the accuracy of the test equipment used. To emphasize the need for an accurate extensometer if satisfactory results are to be obtained, attention is called to the very gradual departure from the apparent proportionality between stress and strain in many of the curves for the "stain-resisting" and high-speed steels (Fig. 9). In such cases, the numerical values of proportional limit would be much higher when using an extensometer reading directly to only 0.001 in. than when using the very accurate equipment shown in Fig. 10 with which the stress-strain curves referred to were obtained. Likewise, at high temperatures, the total deformation at the proportional limit may be of the order of a few ten thousandths of an inch, as shown in Figs. 8 and 9. In recognition of this situation, and to better study the relations between long-duration and short-time tests, the extensometer used in work previously reported by the author, and described several years ago(13), has been replaced by this more accurate one. It is quite similar in details of construction to the modified Martens' extensometer recently described by Wilhelm(1) except that the scale and mirrors have been replaced by the prisms and auto-collimator (the latter is not shown in Fig. 10) of the Tuckerman optical lever system(14). The smallest direct reading on the vernier in the collimator is equal to approximately four millionths of an inch and permits detection of very small departures from the apparent proportionality between stress and strain, provided suitable loading equipment and temperature control are available.

Above the temperatures at which strain hardening is observed, the maximum allowable stress (load permitting long life with freedom from deformation) is equivalent to the "limiting creep stress" referred to by other investigators, and is approximated by the proportional limit of the tension tests at corresponding temperatures. Within the temperature range in which strain hardening is observed the proportional limit also approximates the load permitting long life with freedom from deformation but is below the "limiting creep stress" which includes only consideration of long life.

The need for accurate and sensitive test equipment, emphasized for rapid approximation of load-carrying ability in either fixed-load or tension tests, applies also to flow tests of long duration. Thus far no direct evidence is available to show whether flow actually ceases after strain hardening or whether it continues at very much reduced rates; likewise the question arises whether the rate of flow can ever be zero at temperatures above the range in which strain hardening is observed or whether it occurs at very low velocities even under low loads. The answer to these questions at this time is perhaps of academic interest rather than of practical importance, for in either case metals have a useful load-carrying ability. However, it must be clear from the character of the described relations that the numerical values of load-carrying ability, "limiting creep stresses" or other factors will be dependent upon the accuracy and sensitivity of the test equipment and that this applies to the long-duration tests as well as to determination of stress-strain relations in the tension tests. Because of such conditions the question may properly be raised as to whether accurate determination of stress-strain relations in the comparatively rapid tension tests will not be more valuable, at least, when more study has been given to their interpretation, than so-called "creep" tests made with comparatively inaccurate equipment.

It will perhaps be well to emphasize in this connection that such terms as "cessation of flow," "freedom from deformation," etc., which have been used throughout this report refer to an order of change detectable with the equipment available and do not imply absolute cessation or freedom from flow.

Comparisons of the Steels Tested:

Comparisons of the high-temperature properties of the three steels show some interesting features both with respect to load-carrying ability and resistance to oxidation in air.

The quenched-and-tempered "stain-resisting" steel is superior in loads permitting long life with freedom from deformation to the hot-rolled low-carbon and high-speed steels at atmospheric temperatures but is inferior to the latter between 1100 and 1350° F. (595 and 730° C.). However, it has much better resistance to oxidation in air, particularly in the neighborhood of 1350° F. (730° C.) than the high-speed steel, which in turn is better in this respect than the low-carbon steel.

The load-carrying ability of the "stain-resisting" steel from the standpoint of long life and freedom from deformation is practically nil at 1350° F. (730° C.) and about equal to the plain carbon steel at 1100° F. (595° C.); it is, however, the best at lower temperatures.

As shown by these results, superior chemical stability at high temperatures (oxidation in air in this case) does not necessarily imply superiority in load-carrying ability.

SUMMARY AND CONCLUSIONS

The described tension and flow tests on the three steels may be summarized as follows:

1. Under a fixed total load in tension at approximately constant temperature, the total flow producing fracture takes place in three steps, the magnitude and importance of which vary with the applied load, temperature and material.

2. These three stages of flow are designated (1) an initial flow; (2) a secondary flow; and (3) a final flow. As the constant applied load is increased, the initial flow and the rate of flow in the second period increase and the life of the steels decreases. The final rapid flow is the result of stress concentration brought about by decrease in cross-section accompanying previous elongation.

3. When the logarithm of the life is plotted against the load, the curves obtained show evidence of a bend or "knee" similar to that observed in "stress - log cycle diagrams" of fatigue tests at atmospheric temperatures. This bend appears to be shifted to longer life as the temperature is raised in a given alloy or, in comparison of the three steels at a given temperature, it is at the longest life in the one which has the lowest load-carrying ability.

4. An important effect of temperature increase is to reduce strain-hardening ability which (within the temperature range studied) is greatest in each steel at ordinary temperatures; its rate of decrease and temperature of disappearance vary in the three steels. As a result of these features, the principal factor governing maximum allowable stress varies with temperature and the type of service. Above the temperatures at which strain hardening is observed, the maximum allowable stress, which is taken as the load permitting long life with freedom from deformation, is equivalent to what has been called the "limiting creep stress" by other investigators and is approximated by the proportional limit of the tension tests at corresponding temperatures. Within the temperature range in which strain hardening is observed, the proportional limit also approximates the load permitting long life with freedom from deformation but is below the "limiting creep stress" which includes only consideration of long life.

5. There is recorded in this report the adaptation to high-temperature tension tests of the Tuckerman-Martens optical extensometer

and a brief discussion is given of the dependence of results in both flow tests and tension tests upon the accuracy of the test equipment used.

Acknowledgments.—Acknowledgment is made to H. C. Cross and A. A. Peterson, laboratory assistants, for carrying out the tests on the two special steels; to W. A. Tucker, laboratory assistant, for tests of the carbon steel; to L. B. Tuckerman, Senior Scientist, for suggestions and assistance in construction of the modified Martens extensometer, and to R. V. Campbell, laboratory mechanic, who made this instrument.

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(3) *Proceedings*, International Congress for Testing Materials, Vol. 2, Part I, p. 1 (1900).

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(5) *Journal*, Iron and Steel Inst., 106, p. 103 (1922).

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(11) *Mechanical Engineering*, December, 1925.

(12) *The Metal Industry* (New York) January, 1926, p. 7.

(13) U. S. Bureau of Standards *Technologic Paper No. 219* (1923).

(14) L. B. Tuckerman, "Optical Strain Gages and Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 602 (1923).

DISCUSSION

MR. P. G. McVETTY[1] (*presented in written form*).—The importance of determining the limits of safe working stresses is generally recognized and this paper by Mr. French is a valuable contribution to the literature. The increasing use of metals at elevated temperatures has made it necessary to determine these limits under conditions as near as possible to those of service. Working stresses determined by the long-time high-temperature test are therefore applicable to sustained tension loading, but it has not yet been proved that this test is conclusive for high-temperature applications involving stresses other than pure tension or stresses which vary either in amount or in direction. Further development may establish some method of correlation among the several types of high-temperature tests, but in the present state of our knowledge the data here presented may be used with assurance of safety only in applications involving sustained tension loading. **Mr. McVetty.**

Reference to Figs. 1 and 5 of the paper shows that a choice of stresses is offered, depending upon the conditions of application. It is the purpose of the writer to discuss the field of strain hardening shown in these figures. This field is bounded by two curves called "maximum stress for long life with deformation" and "maximum stress for long life without appreciable deformation."

For the purposes of the designer it appears that the choice of working stresses for high-temperature applications is entirely a matter of allowable deformation within the expected life of the material. For some applications, the amount of deformation is unimportant providing that fracture does not occur. Other applications may restrict the total deformation to a definite limit depending upon the relation of the stressed member to those adjacent to it. The latter case includes the limit called by the author "long life without appreciable deformation," for it can be shown that there is a definite deformation for any stress, however small, and the determination of this lower limit depends upon the definition of the term "appreciable deformation." It seems important also to define what is meant by "long life," since the term is relative and might imply a few months in some applications and many years in others.

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Mr. McVetty. The statement of the author that strain-hardening ability is greatest at atmospheric temperature is open to some question. Sauveur and Lee[1] have found that the maximum increase in strength and hardness resulting from strain hardening occurs in the blue-heat range of the material rather than at normal temperature. On that basis, the upper boundary of the strain-hardening range of 0.24-per-cent carbon steel should have the same general shape as the tensile strength curve, decreasing to a minimum at about 100° C. (212° F.), then rising to a maximum at about 300° C. (572° F.), then decreasing rapidly to the intersection with the proportional-limit curve. It is expected that further tests will confirm this conclusion, but it will be necessary to conduct tests over much longer periods of time than any that have been reported to make sure that the deformation will not, within the life of the material, cause sufficient reduction in cross-section to produce fracture.

One of the most important points on the diagrams shown in Fig. 5 is the intersection of the two life curves. The temperature at which this intersection occurs varies with the material, and for stain-resisting steel is considerably higher than for 0.24-per-cent carbon steel. In Fig. 9, the slope of the elastic line for stain-resisting steel at 800° F. (425° C.) appears to be greater than the slope at the two lower temperatures. The writer is not familiar with the characteristics of material of this particular analysis, but in general, the modulus of elasticity decreases as the temperature is increased. A slight decrease in the slope of this line would tend to raise considerably the selected value of proportional limit at 800° F. (425° C.) and remove the dip at this temperature in the proportional limit curve in Fig. 5.

The determination of the lower limit of the strain-hardening range requires the utmost refinement of strain measurement. In tests previously reported,[2] the stretch in the long-time tests was measured to an accuracy of four-millionths of an inch, using the same type of extensometer as in the short-time tests. The order of accuracy of this apparatus was the same as that of the Tuckerman-Martens extensometer recently adopted by Mr. French. With this equipment, the writer experienced considerable difficulty due to occasional accidental disturbance of the Martens telescopes during the progress of long-time tests. In continuing the joint investigation with Mr. N. L. Mochel for the South Philadelphia Works, we have, therefore, abandoned the use of Martens telescopes on all of our long-time testing machines, which have now been equipped with extensometers **Mr. McVetty.** which read to approximately one-millionth of an inch. A specially designed projection apparatus rigidly attached to the testing machine projects the focussed image of the cross-wires upon a scale on the wall about 15 ft. from the mirrors. This arrangement has been found to be very satisfactory in service and it eliminates the uncertainty which always exists when an irregularity in the flow curve follows an accidental movement of the extensometer. The type of curves obtainable with this apparatus are shown in Figs. 2, 3 and 4 of this discussion.

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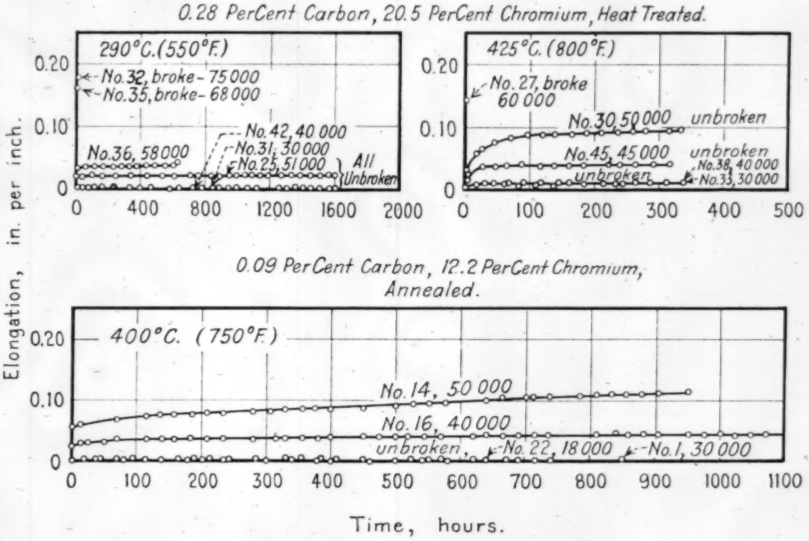
[1] Albert Sauveur and D. C. Lee, "The Influence of Strain and of Heat on the Hardness of Iron and Steel," *Journal*, Iron and Steel Inst., Vol. CXII, 1925.

[2] T. D. Lynch, N. L. Mochel and P. G. McVetty, "The Tensile Properties of Metals at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 5 (1925).


0.28 PerCent Carbon, 20.5 PerCent Chromium, Heat Treated.

0.09 PerCent Carbon, 12.2 PerCent Chromium, Annealed.

FIG. 1.—Flow of Chromium Steels Under Sustained Tension Loads at Various Temperatures.

Referring to Fig. 3 of Mr. French's paper, the flow tests of high-chromium steel at 550° F. (290° C.), indicate practically no change of length after the first few hours. The fact that tests were discontinued after 1600 hours leads us to the conclusion that the amount of further extension to be expected is negligible. Apparently, the tests at 800° F. (425° C.) were discontinued after about 350 hours. Referring to Fig. 5, it is evident that the author considered 58,000 lb. per sq. in. at 550° F. (290° C.) and 50,000 lb. per sq. in. at 800° F. (425° C.), as safe stresses if deformation is allowable. In the same Fig. 3, test No. 47 at 1100° F. (595° C.), and test No. 14 at 1350° F. (730° C.) broke after 800 hours of test. This raises the question of what would have happened if test No. 36 at 550° F. (290° C.) and

Mr. McVetty. test No. 30 at 800° F. (425° C.) had been continued, and whether or not the stresses shown in Fig. 5 do give long life.

In the accompanying Fig. 1 the two upper charts of Fig. 3 have been reproduced for comparison with similar curves obtained by the writer in testing an annealed stainless iron. The materials and temperatures are slightly different but the long-time test data should be comparable. This particular material is used for the comparison because it seems desirable to base a discussion of test methods and interpretation of results upon material showing the maximum uniformity among individual test pieces. When plotted to the same

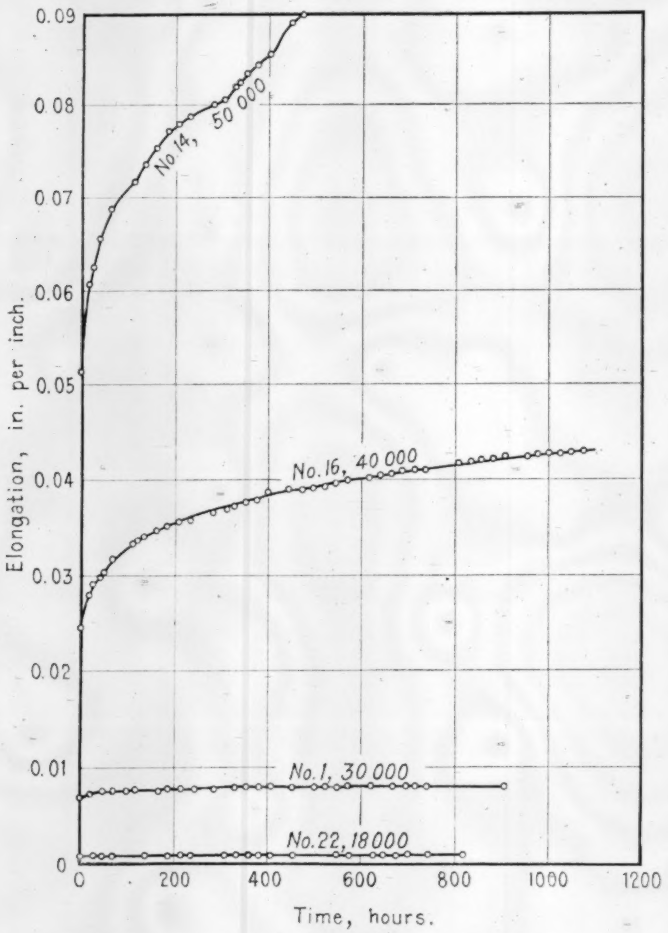


FIG. 2.—Flow of Annealed Stainless Iron at Various Stresses, Tested at 400° C. (752° F.).

Mr. McVetty.

scale, the curve for test No. 14 at 50,000 lb. per sq. in. and 400° C. (750° F.) is almost identical with the author's curve for test No. 30 at the same stress and a temperature of 425° C. (800° F.). The continuation of the curve for test No. 14 to 800 hours indicates a slight increase in extension, but the rate of increase is not greater than is shown by the author for test No. 30. We might then say that a stress slightly less than 50,000 lb. per sq. in. would give long life with deformation but we are continuing both the 50,000 and 40,000 lb. per sq. in. tests to prove whether or not this is true.

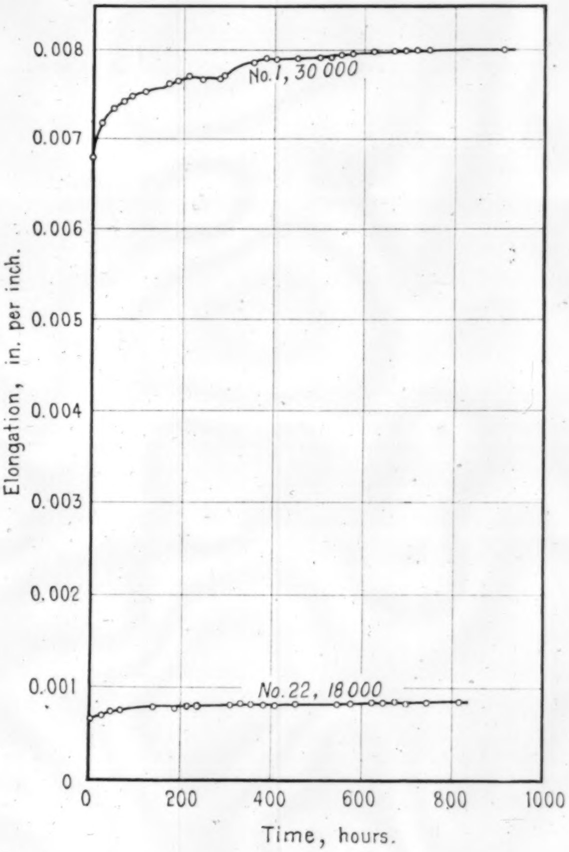


FIG. 3.—Flow of Annealed Stainless Iron at Various Stresses, Tested at 400° C. (752° F.).

Figure 2 shows the same test data plotted to a scale of ordinates 20 times as great as in Fig. 1. The peculiar variations now revealed in the curve for a stress of 50,000 lb. per sq. in. are probably due to

Mr. McVetty. progressive strain-hardening, modified by the effect of decreasing cross-section and increasing stress. In general, this form of curve is characteristic of a test which ultimately reaches fracture. At this scale, the 40,000 lb. per sq. in. test is also stretching at a fairly rapid rate, but the 30,000 and 18,000 lb. per sq. in. tests appear to have reached a constant length.

Figure 3 shows the same test data plotted to a scale 200 times as great as in Fig. 1. In this chart, the test at 30,000 lb. per sq. in. has assumed the shape which may lead to ultimate fracture and only the 18,000 lb. test shows an apparent constant length. The latter stress is 1000 lb. per sq. in. below the proportional limit at this temperature.

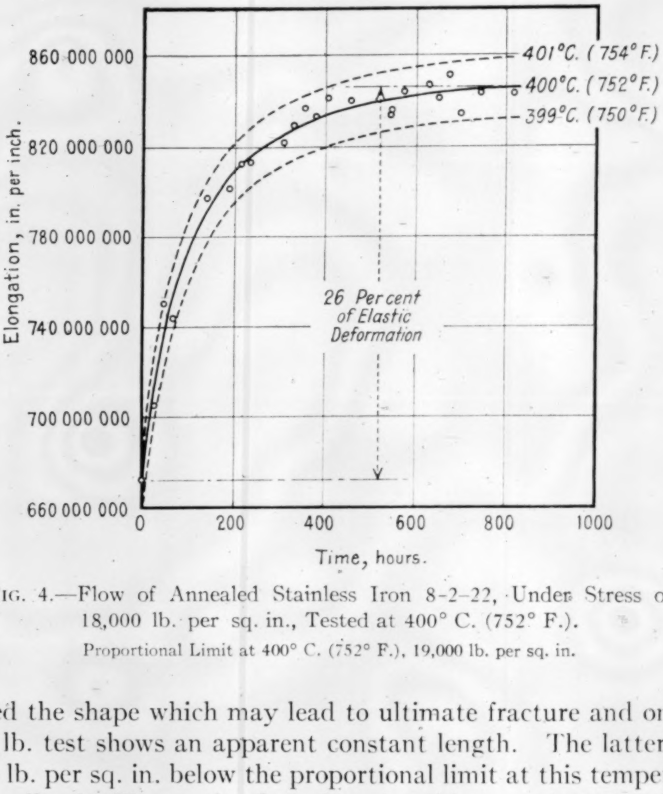


FIG. 4.—Flow of Annealed Stainless Iron 8-2-22, Under Stress of 18,000 lb. per sq. in., Tested at 400° C. (752° F.).

Proportional Limit at 400° C. (752° F.), 19,000 lb. per sq. in.

Finally, in Fig. 4, the flow data for this test at 18,000 lb. per sq. in. have been plotted to a scale 5000 times as great as in Fig. 1. The dotted lines show the effect of a temperature variation of 1° C. either way, proving that the temperature control is within these limits. At this magnified scale, some scatter of the points must be expected, but it is significant to note that even this enormous change of scale has not materially affected the conclusions which might have been drawn from Fig. 3.

Mr. McVetty.

The creep in the first 800 hours of test amounts to 26 per cent of the elastic deformation at this stress. From the data so far available it appears that some deformation of this order is to be expected at elevated temperatures even with low stresses. This must be considered in the interpretation of the term "without appreciable deformation." It is evident from Fig. 4 that further refinement of strain measurement is useless unless methods of temperature control are improved. It is the opinion of the writer that any increase in length after that shown in the first 800 hours of this particular test may be considered negligible. Even if prolonged at the same rate of stretch as in the last 200 hours it would take nearly six years to produce a further change of 0.001 in. per inch. The form of the curve indicates a decreasing slope, so that either this time would be considerably extended or flow would stop completely within a relatively short time. This confirms the conclusion advanced a year ago[1] that a stress corresponding to, or slightly lower than, the proportional limit can be carried by this material indefinitely without causing continued extension.

A similar analysis may be applied to stresses within the strain-hardening range. In this case, however, definite life for the material and a definite maximum allowable deformation must be decided upon at the start. It will then be possible to determine by means of the long-time test the proper working stress to be used.

The establishment for any type of service of a maximum allowable total deformation within a definite span of life of the material appears to be a more logical basis for design than the requirement of absence of appreciable deformation.

In conclusion, the data now available indicate that:

1. An accurately determined proportional limit at the working temperature may be considered as the maximum safe stress at that temperature for the steels which have been tested.
2. The long-time test under working conditions of stress, temperature and type of loading should be used to determine the amount of deformation to be expected within the desired life of the material.
3. Both of the above are dependent upon a high degree of refinement of strain measurement and temperature control.
4. Caution should be used in applying conclusions drawn from tests representing sustained tension loading at high temperature to problems of design in which the stress varies in amount or direction or the stress is not pure tension.

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[1] T. D. Lynch, N. L. Mochel and P. G. McVetty, "The Tensile Properties of Metals at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 5 (1925).

Mr. French. MR. H. J. FRENCH (*author's closure by letter*).—I am indebted to Mr. McVetty for his interesting confirmation of the results described in this paper. The majority of the questions he has raised appear to be more directly related to terminology than to the behavior of steels under sustained loads at various temperatures, and probably arise from the fact that space limitations made it necessary to condense to a high degree the discussion of results. Clearer conceptions and the answer to most of the questions can be obtained by referring to the author's initial contribution to the study of "creep" referred to in item (7) of the assembled references in the paper.

With reference to the effect of temperature on the strain hardening of steels, Mr. McVetty has apparently failed to distinguish between what may be called capacity for strain hardening or strain-hardening ability and the rate of strain hardening. These effects are discussed by Jeffries and Archer in their book entitled "The Science of Metals," page 185, as follows:

> "The time required for this increase in resistance (to slip) is less as the temperature rises, so that at about 250 to 300° C. (blue heat) the change is practically instantaneous and occurs during the progress of any ordinary deformation such as a tensile test. . . .
>
> "This statement applies to deformations effected within a short period of time, such as an ordinary tensile test. If the deformation which takes place during the tensile test at room temperature could be effected very slowly so that there was time for elastic recovery during the test, it would be expected that the tensile strength would increase to an extent comparable with the increase obtained at 'blue heat.'"

The rate of strain hardening comes more into play in the customary tensile test in which the tensile strength is higher at blue heat than at atmospheric temperatures as is shown in Fig. 5 of the paper. Where sufficient time is given, as in the "flow tests," an indication is obtained of the strain-hardening capacity, and as indicated in Fig. 5, this is greater at atmospheric temperatures than at blue heat for the materials tested.

COMPRESSIVE STRENGTH AND DEFORMATION OF STRUCTURAL STEEL AND CAST-IRON SHAPES AT TEMPERATURES UP TO 950° C. (1742° F.)[1]

By S. H. Ingberg[2] and P. D. Sale[3]

Synopsis

This paper presents the results of about 100 tests conducted at the U. S. Bureau of Standards on typical rolled steel shapes and one hollow round cast-iron shape. The specimens are heated within a horizontal furnace with end compensating heating coils to neutralize end losses, the temperature uniformity over a 6-in. gage length being generally within a few degrees Centigrade. Loads are applied by means of a hydraulic piston with suitable calibrated load-indicating devices. The deformations are measured over a 6-in. gage length, using the suspended wire method. The tests have been confined to structural steels of the soft and medium grades and to gray cast iron.

For symmetrical sections the results give a considerable rise in strength over the cold strength in the region of 250° C. (482° F.). Beyond 400 to 500° C. (752 to 932° F.) there is a decided decrease in strength, failure under working loads ordinarily applied being in the region of 550 to 700° C. (1022 to 1292° F.). In most of the tests the specimen is heated up to a given temperature with measurement of expansion under no load, and then loaded to failure. In some of the tests a constant load is applied at the beginning of the test and failure obtained by increasing the temperature with the specimen under constant load. A study on effect of duration of load on ultimate strength at various temperatures is included, the durations from 15 minutes to 4 hours being within the limits of interest from the standpoint of fire resistance of building members. These gave failures or deformations equivalent to failure in large building members at loads as low as one-half of those required to cause failure of the 10½-in. specimens for short time application of load. These tests were confined to the temperature range 595 to 705° C. (1103 to 1301° F.).

There is also included a brief series on effect of length on strength at temperatures near 600° C. (1112° F.). Reducing the 10½-in. length by one-half increased the compressive strength 17 per cent, and reducing the length by three-fourths increased the load 22 per cent. The average results with light angles and channels 10½ in. long were nearly the same as for larger built-up steel columns 12 ft. 8 in. long, previously tested.

The present series of tests to determine the effect of temperature on the compressive strength and deformation of structural steel and cast iron was planned primarily to give information applicable to

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[1] Published by permission of the Director of the U. S. Bureau of Standards, Department of Commerce, Washington, D. C.

[2] Physicist, U. S. Bureau of Standards.

[3] Associate Physicist, U. S. Bureau of Standards.

TABLE I.—PROPERTIES OF MATERIALS AND SPECIMENS.

Material	Shape	Elements of Structural Shapes						Tensile Properties at Room Temperature, average values for 3 to 7 tests from representative parts of each section						Chemical Analysis					
		Nominal Weight, lb. per ft.	Nominal Area, sq. in.	Minimum Metal Thickness, in.	Least Radius of Gyration, r, in.	Length, l, in.	Slenderness Ratio, $\frac{l}{r}$	Proportional Limit, lb. per sq. in.	Yield Point,[e] lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Modulus of Elasticity, lb. per sq. in.	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Other Elements, per cent
Cast iron.......	Hollow round.......... Outside diameter $1\frac{1}{2}$ in. Inside diameter $\frac{1}{2}$ in...	5.5	1.61	0.50	0.395	9.25[a]	23.4	No tension tests. Transverse test gave computed ultimate extreme fiber stress of 60,000 lb. per sq. in.						0.42 combined 3.10 graphitic	0.37	0.715	0.115	1.580	....
Structural steel[b]...	3-in. I-beam, $5\frac{1}{2}$ lb.....	5.5	1.63	0.17	0.53	10.5	19.8	35 000	43 700	68 700	36.6	63.4	28 300 000	0.21	1.15	0.016	0.034	0.025	Cr. 0.06 Cu. 0.18
Structural steel[c]...	3-in. I-beam, $5\frac{1}{2}$ lb.....	5.5	1.63	0.17	0.53	10.5	19.8	33 200	42 000	66 900	46.3	50.3	29 200 000	0.21	0.81	0.020	0.052	0.030	....
Structural steel[d]...	3-in. I-beam, $5\frac{1}{2}$ lb.....	...	....	....	....	....	....	22 300	34 200	60 200	38.3	45.9	29 800 000	0.21	0.81	0.020	0.052	0.030	....
Structural steel....	4-in. channel, $7\frac{1}{4}$ lb....	7.25	2.13	0.32	0.46	10.5	22.8	32 200	39 700	61 700	35.2	62.2	29 750 000	0.21	0.58	0.011	0.044	0.050	....
Structural steel....	4-in. channel, $5\frac{1}{4}$ lb ...	5.25	1.55	0.18	0.45	10.5	23.4	37 300	48 700	63 800	28.3	53.9	29 625 000	0.17	0.56	0.036	0.055	0.040	....
Structural steel....	$2\frac{1}{2}$ by $2\frac{1}{2}$ by $\frac{1}{2}$-in. angle	7.7	2.25	0.50	0.47	10.5	22.3	32 900	38 100	59 800	33.8	62.1	31 000 000	0.20	0.34	0.018	0.063	0.050	....
Structural steel....	3 by 3 by $\frac{1}{4}$-in. angle...	4.9	1.44	0.25	0.59	10.5	17.8	32 100	39 900	58 400	37.5	60.3	28 700 000	0.21	0.57	0.014	0.035	0.020	....
Structural steel....	2-in. solid round.......	10.7	3.14	2.00	0.50	10.5	21.0	29 300	33 000	60 000	38.3	56.1	29 500 000	0.20	0.38	0.013	0.032	0.010	....

[a] Clear distance between $\frac{5}{8}$-in. end flanges.

[b] High manganese content.

[c] Medium manganese content, not annealed.

[d] Medium manganese content, annealed.

[e] By drop of beam and at times by dividers.

NOTE.—Tension specimens were rectangular flats from $\frac{1}{2}$ to $1\frac{3}{4}$ in. wide at the reduced section and were cut from webs and flanges. Rounds from $\frac{1}{4}$ to $\frac{1}{2}$ in. in diameter were cut from the roots. The length of the reduced section was about 4 in. for the flats and $2\frac{1}{2}$ in. for the rounds.

fire resistance of supporting members in buildings. Under fire conditions the temperature rise, particularly of unprotected members, is quite rapid but the duration of high temperature seldom extends beyond a few hours. Fire tests of full-size building members, such as columns and floors, are conducted to give information on the fire resistive properties of the construction and do not generally yield accurate data on the properties at high temperatures of the materials concerned. Usually more than one material is present in one member, such as a column, which may have a load-bearing metal core embedded in or covered by one of the several kinds of heat-resistant protective materials that may also take portions of the load applied. In fire tests, the temperature variation across the metal section and along the length of the member is generally large. In the present tests, typical shapes of structural steel and one of cast iron were heated with a degree of temperature uniformity over a given length that would give conditions more favorable for the determination and correlation of strength, deformation and temperature data.

MATERIALS AND SPECIMENS

The properties of the materials and specimens tested are given in Table I. The steel specimens were standard rolled shapes, the sizes of which were governed by the limits of practicable diameter and length for the heating furnace. The regular length for tests herein reported was $10\frac{1}{2}$ in. Tests were also made of similar specimens of one-half and one-fourth this length and further tests with solid rounds are planned that will give slenderness ratios from 21 to 144. The results of the test at room temperature of the 2-in. solid round are given in the table and Fig. 3 for comparison with results of rolled sections of similar slenderness ratio. All specimens of a given shape were cut from one bar length.

The results of tension tests and chemical analysis indicate for the angle and channel sections an average grade of structural carbon steel. The tension tests were made on samples cut from representative portions of each section. The I-beam sections gave higher ultimate strengths due apparently to a higher manganese content. The one with 1.15 per cent of manganese was tested throughout the temperature range without previous annealing. To obtain material more nearly comparable with that of the other sections another length of I-beam was obtained, analysis of which showed 0.81 per cent of manganese. This had a tensile strength at room temperature of 66,900 lb. per sq. in. After annealing at about 750° C. (1382° F.) the tensile strength was reduced to 60,200 lb. per sq. in., which is near the 60,900 lb. per sq. in. average for the angle and channel sections. Tests of all other sections at room and higher temperatures were made without initially annealing the specimens.

The chemical analysis of the cast iron indicates an ordinary gray iron somewhat high in sulfur.

TESTING EQUIPMENT AND METHODS

Figure 1 is a general view of the equipment showing the main furnace *B* rolled back to show the specimen *A* in place and the end compensating heating coils *C* that project 2 in. over the ends of the specimen. Further compensation for heat losses at the ends of the specimen is obtained with the coils *D* one on each end bearing block. The temperature gradient is further reduced by the clay or porcelain end bearing blocks *E* and by operating the main furnace with the heating current applied only over the outer quarters of its length, the wiring and connections being arranged for independent control of the heating coils over the middle half of the main furnace tube. Six independent rheostat controls are provided for the different portions of the heating system. These details were all found necessary if a temperature uniformity within a few degrees Centigrade

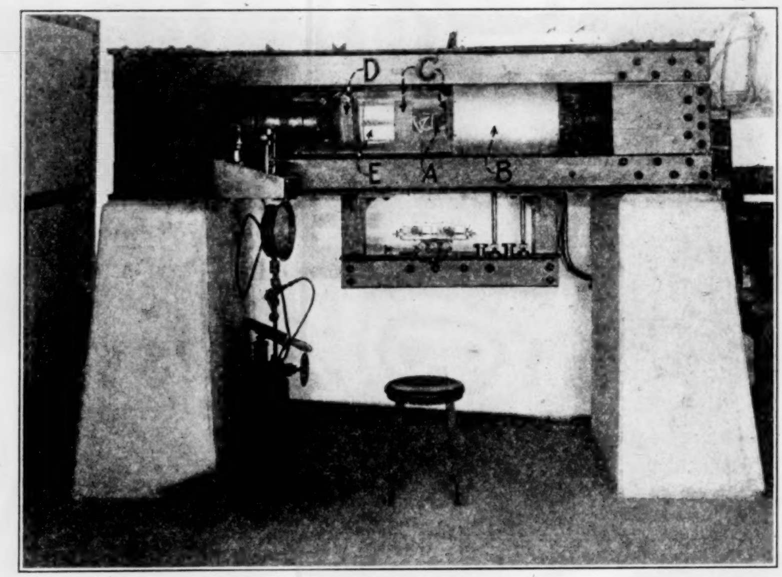

FIG. 1.—View of Equipment.

was to be maintained over the 6-in. (150-mm.) gage length. This is considered essential as a basis for temperature, expansion, strength and deformation determinations, since such a degree of temperature uniformity and consequent absence of any decided heat transfer both across the section and over a considerable portion of the length of the specimen are necessary conditions for valid determinations.

The load is applied to the specimen axially by hydraulic pressure through adjustable bearing blocks, all of which, inclusive of the furnaces, are counterweighted to obviate bending stresses in the test specimen. The hydraulic pressure is measured by a fluid pressure scale, the readings of which are evaluated in terms of load on the specimen by calibrations with elastic springs and bars. The accuracy of the load applications is estimated to be between ±250 and ±500 lb., the maximum load capacity as limited by the strength of the restraining frame being 264,000 lb. (120,000 kg.).

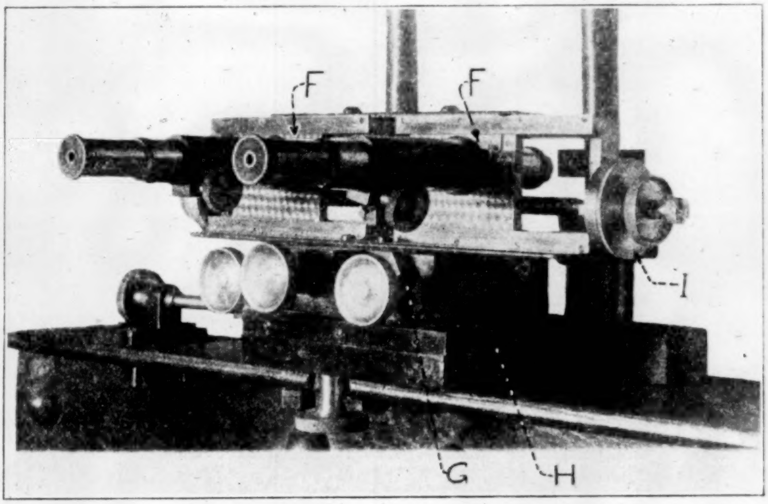

FIG. 2.—Mounting for Microscopes.

For expansion and deformation measurements, suitably formed pegs are screwed into the test specimen at each gage point with fine heat-resistant alloy wires attached at the centroid of the section tested. These wires depend through the furnace protection and brass protecting tubes, with weights attached immersed in oil to dampen vibrations. The microscopes are mounted in micrometer slides *F*, Fig. 2, secured to one transverse pivoted slide, *G*, to permit refocusing the microscopes on the wires without moving the microscopes in their tubes, with consequent possibility of errors due to changes in the line of sight. The longitudinal slide, *H*, permits setting the cross hair of one microscope without moving its slide *F*, the expansion or compressive deformation in the gage length being thus measured directly with the other slide by sighting the microscope mounted in it on the wire suspended from the opposite end of the gage line of the test specimen. The micrometer head *I* is graduated to 0.005 mm. and can be set and readings estimated to the nearest 0.001 mm. or 0.00004 in. The general accuracy of measurement is probably not better than ±0.003 mm. (0.0001 in.) which is equivalent to ±0.00002 deformation per unit of length. The slides *F* have a combined range of 100 mm. (4 in.) but the clearance for the wires within the present protecting tubes limits the total movement to about 9 mm. or 6 per cent of the gage length.

The temperatures were measured with eight iron-constantan thermocouples peened into the metal of the test specimen, four at representative points on the section at the midpoint of the gage length and two at each end, the average temperature in the gage length being obtained from the numerical average of the eight readings. The cold junctions were separately iced and were connected to a potentiometer reading the thermal electromotive forces of the couples to the nearest 0.01 millivolt. These were transposed to temperatures from comparison calibration of the thermocouple wires, the accuracy of the temperature measurements being estimated to be ±1° C. at temperatures up to 300° C. (572° F.), ±2° C. in the range 300 to 600° C. (572 to 1112° F.) and ±3 to 5° C. for higher temperatures.

The tests were conducted either by applying a constant load to the specimen and gradually increasing its temperature until failure occurred, or by heating it under no load up to a given temperature and applying load until failure while holding the temperature constant. The first method is representative of conditions to which building columns are subjected when exposed to fire. When so tested the material expands until a temperature is attained, depending largely on the intensity of the load applied, at which the rate of yielding due to the load is equal to the thermal expansion, and beyond this the resultant deformation is compressive. The results of tests at constant temperature are here reported in greater detail than those conducted at constant load, only the ultimate strength so obtained being included for comparison in Fig. 12.

The failure or ultimate strength is taken as the highest load per unit area that the specimen would support under the test conditions. Failure was accompanied by buckling of the whole specimen or parts thereof such as angle legs or channel and I-beam flanges and webs. Failure was well defined in all tests except for two in the series on effect of duration of loading, which will be noted later.

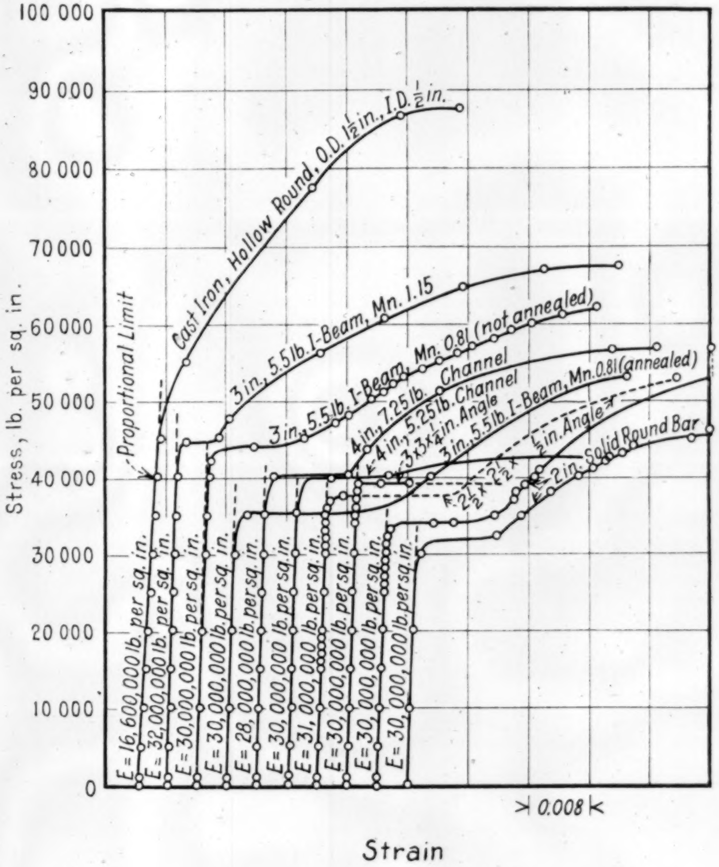

FIG. 3.—Stress-Strain Curves from Compression Tests of Structural Steel and Cast Iron at Room Temperature.

DATA FROM TESTS

In Fig. 3 are shown stress-strain curves from compression tests at room temperature, on each of which is marked the point (Proportional Limit) beyond which the ratio of stress to strain ceases to be constant, also the value of the ratio below this point. The initial ratio of stress to strain, *E*, was determined by the slope of a straight line passing

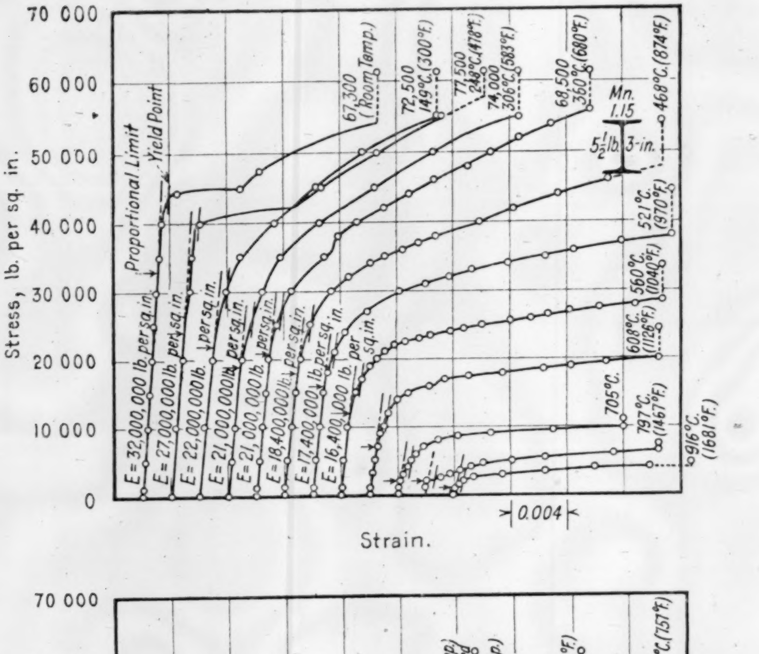

FIG. 4.—Stress-Strain Curves from Compression Tests of Structural Steel I-Beam Sections at Constant Temperatures.

generally through zero and as many experimental points, plotted to large scale, as appeared to satisfy a straight line trend of the data. The yield point is indicated clearly by the curves themselves except in the case of cast iron, which shows no definite point of yield. The ultimate strengths are indicated by the end points of the curves and are higher than the yield point for all specimens except the light angle

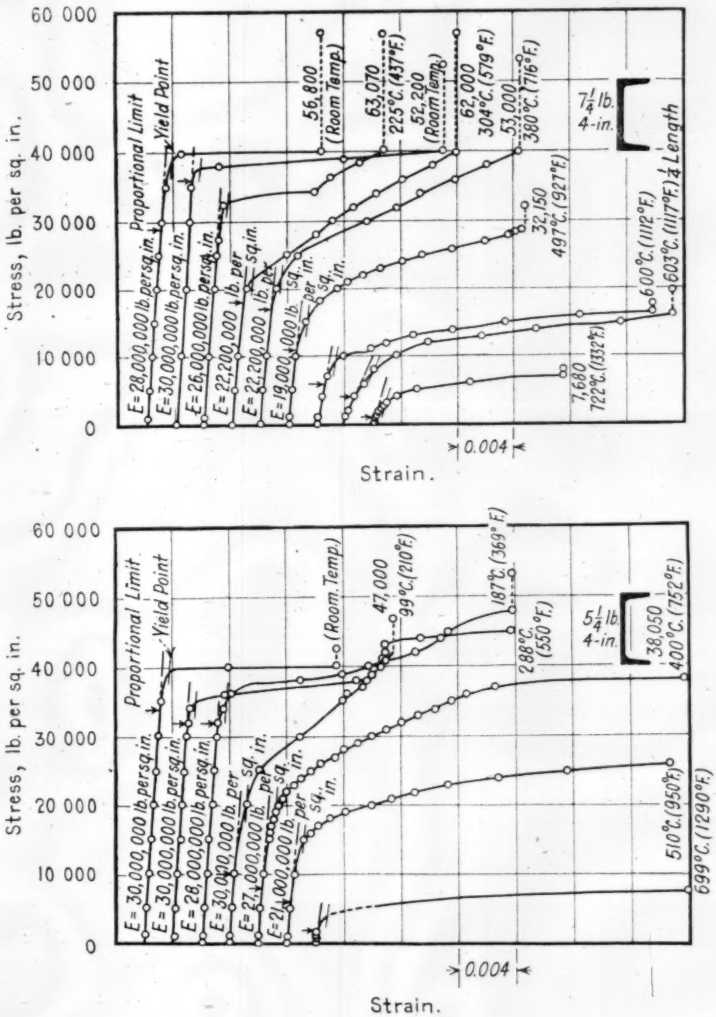

FIG. 5.—Stress-Strain Curves from Compression Tests of Structural Steel Channel Sections at Constant Temperatures.

section. The deformations near the ultimate are not plotted for a number of the end points in Figs. 3 to 6 to avoid interference between curves, or unduly extending the diagrams. In some cases the deformation near the ultimate was greater than could be measured with the clearance available for the wires.

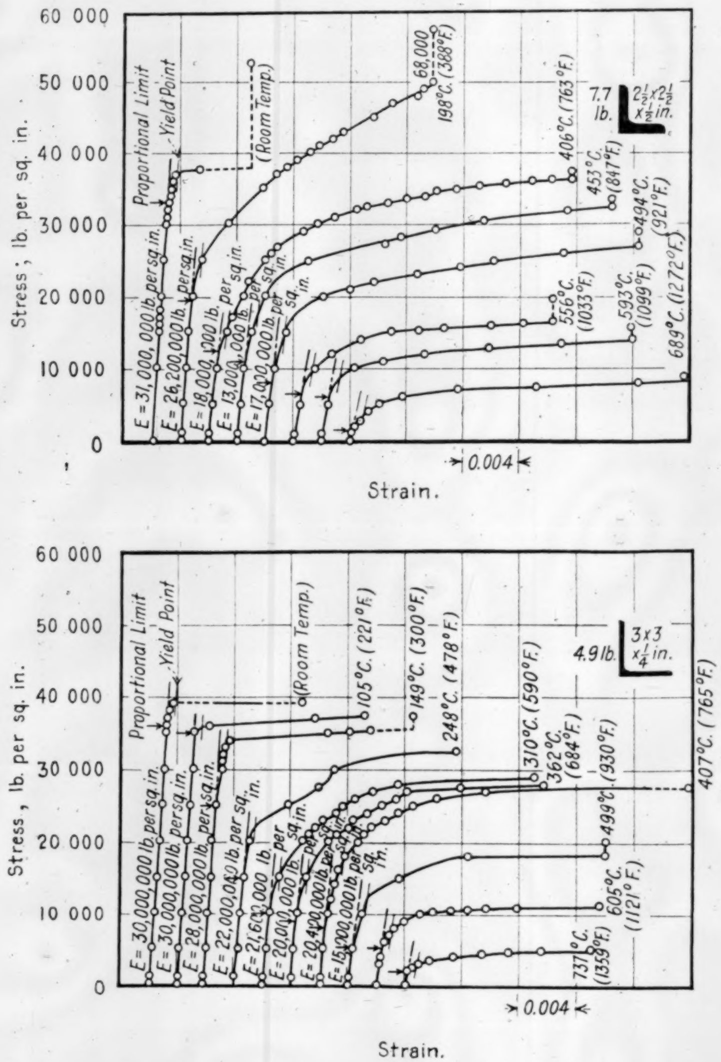

FIG. 6.—Stress-Strain Curves from Compression Tests of Structural Steel Angle Sections at Constant Temperatures.

In Figs. 4, 5 and 6 are shown the stress-strain curves for the steel sections as tested to failure at the given temperatures and in Fig. 7 are shown those for cast iron. Beyond 400 to 500° C. (752 to 932° F.) the proportional limit was not as well defined as at lower temperatures. The yield points were arbitrarily chosen as the stresses at which the unit deformations departed from the initial modulus line, 0.0005 for steel and 0.001 for cast iron. This marks with fair consistency the beginning of a decided change in the ratio of increments of stress to strain. As in Fig. 3 the ultimate strengths are indicated by the end points of the lines.

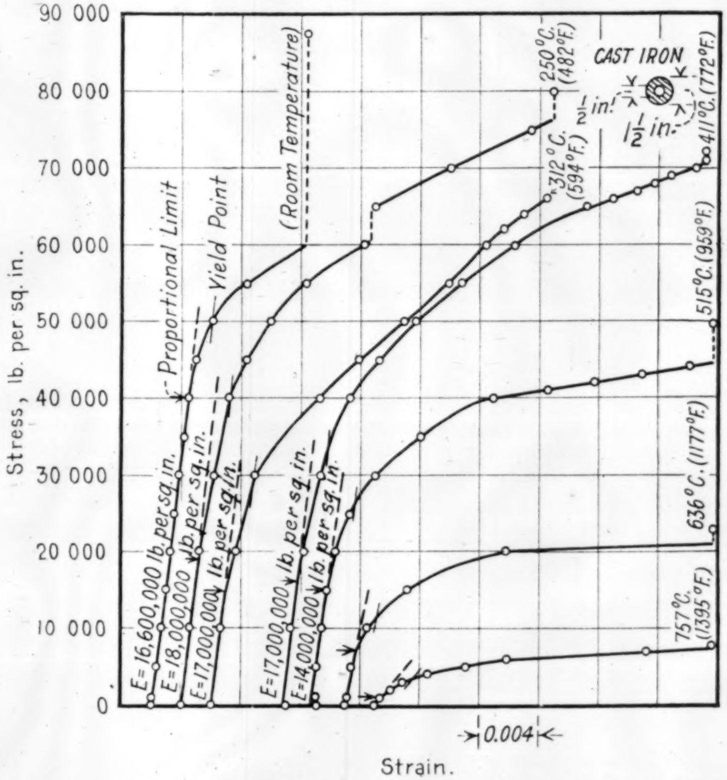

FIG. 7.—Stress-Strain Curves from Compression Tests of Cast Iron at Constant Temperatures.

In Fig. 8 are shown for cast iron the ultimate compressive strength, yield point and proportional limit in tests at constant temperature and in Figs. 9 to 11 the same data are given for structural steel. A marked characteristic is the rise in ultimate strength for all steel sections, except the light angle, in the temperature region near 250° C.

(482° F.) which is within the "blue heat" range. Sections with the higher symmetry of shape or heavier metal thickness attained the greater strengths in this region and at higher temperatures consistently showed a higher strength than the less stable sections. The rapid decrease in strength with temperature beyond 250° C. (482° F.) was not definitely checked until at or a little beyond 700° C. (1292° F.) at temperatures corresponding approximately with the thermal critical point of the steel. There was no rise in yield point or proportional limit near 250° C. (482° F.). Rapid decline obtained after 150 to 200° C. (302 to 392° F.) continuing generally up to 300° C. (572° F.), after which the decline with temperature was less rapid.

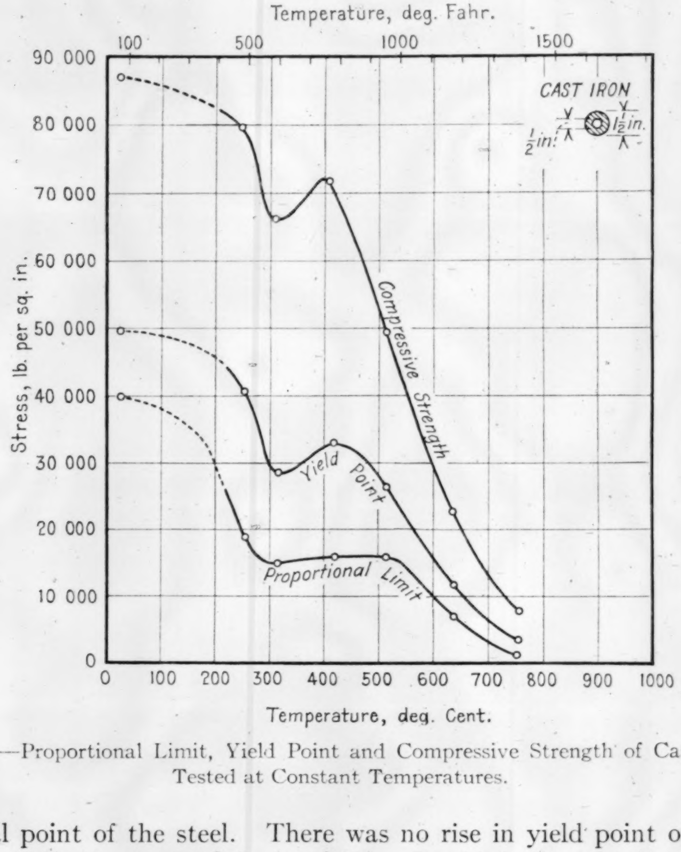

FIG. 8.—Proportional Limit, Yield Point and Compressive Strength of Cast Iron Tested at Constant Temperatures.

For cast iron (Fig. 8) the same general trend of characteristics can be noted except that there was no rise in compressive strength in the region near 250° C. (482° F.). No tests were made at constant temperature above 757° C. (1395° F.), nor at constant load above

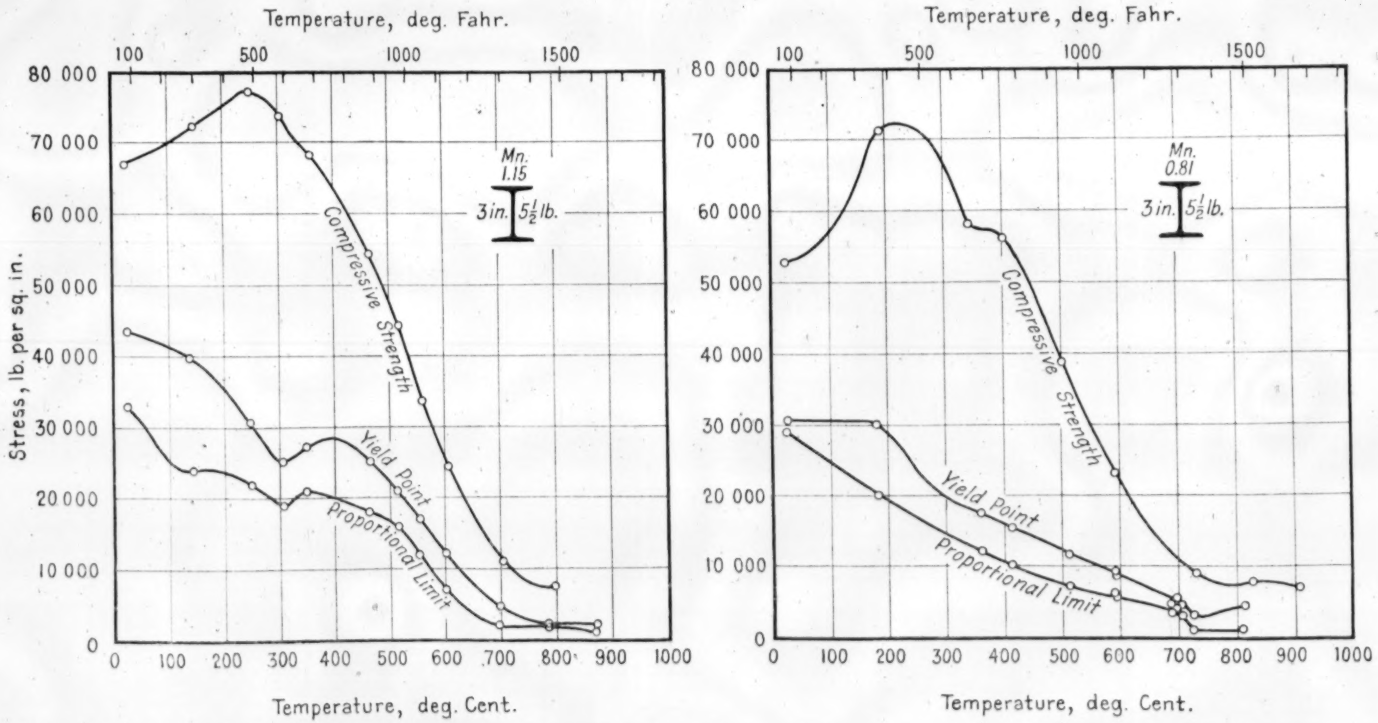

FIG. 9.—Proportional Limit, Yield Point and Compressive Strength of Structural Steel I-Beam Sections Tested at Constant Temperatures.

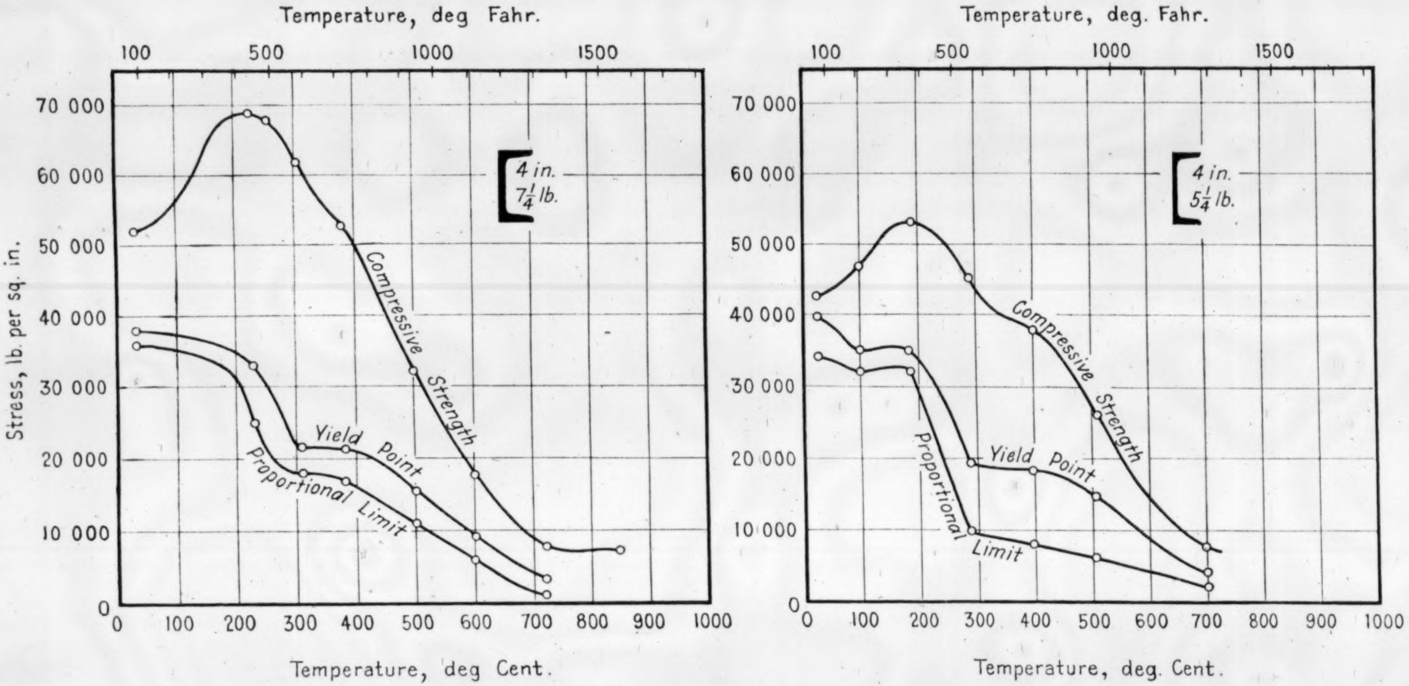

FIG. 10.—Proportional Limit, Yield Point and Compressive Strength of Structural Steel Channel Sections Tested at Constant Temperat ures

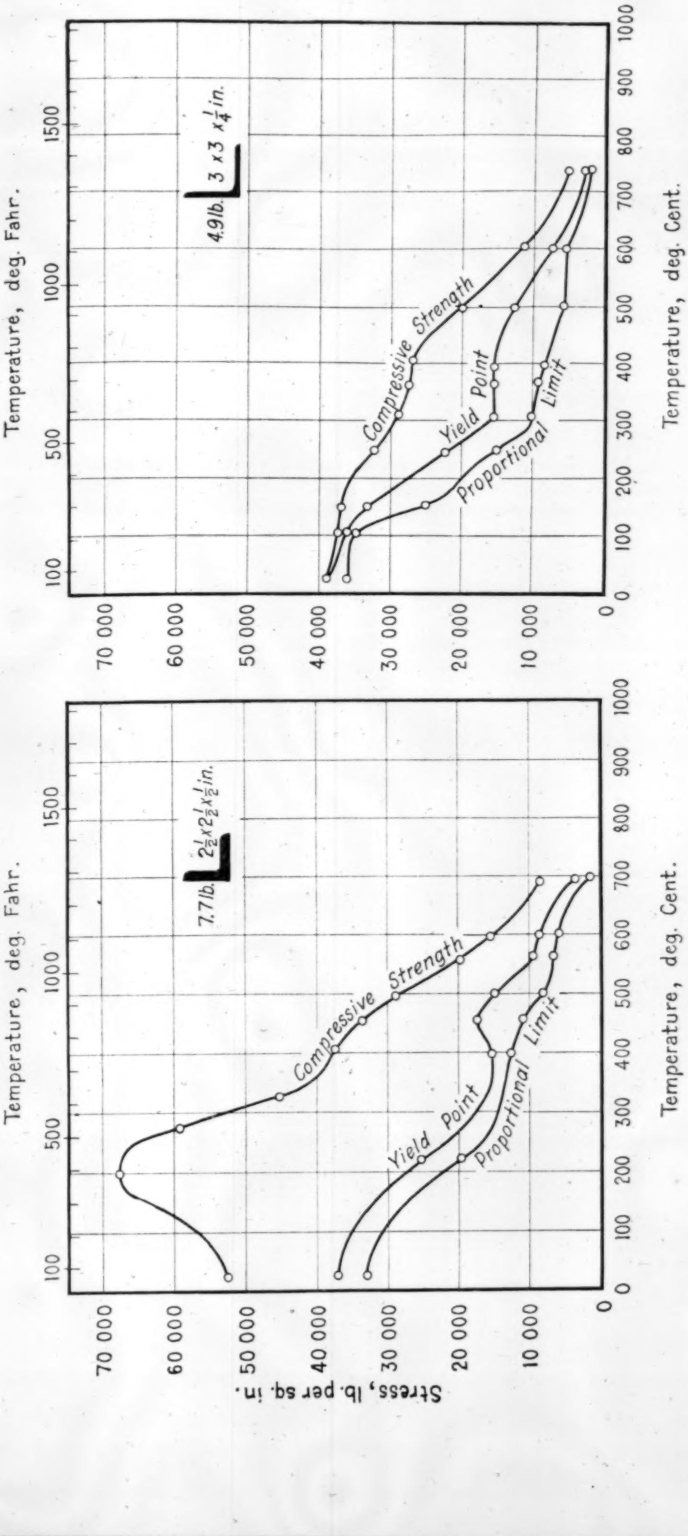

FIG. 11.—Proportional Limit, Yield Point and Compressive Strength of Structural Steel Angle Sections Tested at Constant Temperatures.

790° C. (1454° F.), the strength in this region approximating that of the steel I-beam or heavy channel sections.

In Fig. 12 a comparison on ultimate compressive strength of all sections tested is shown.

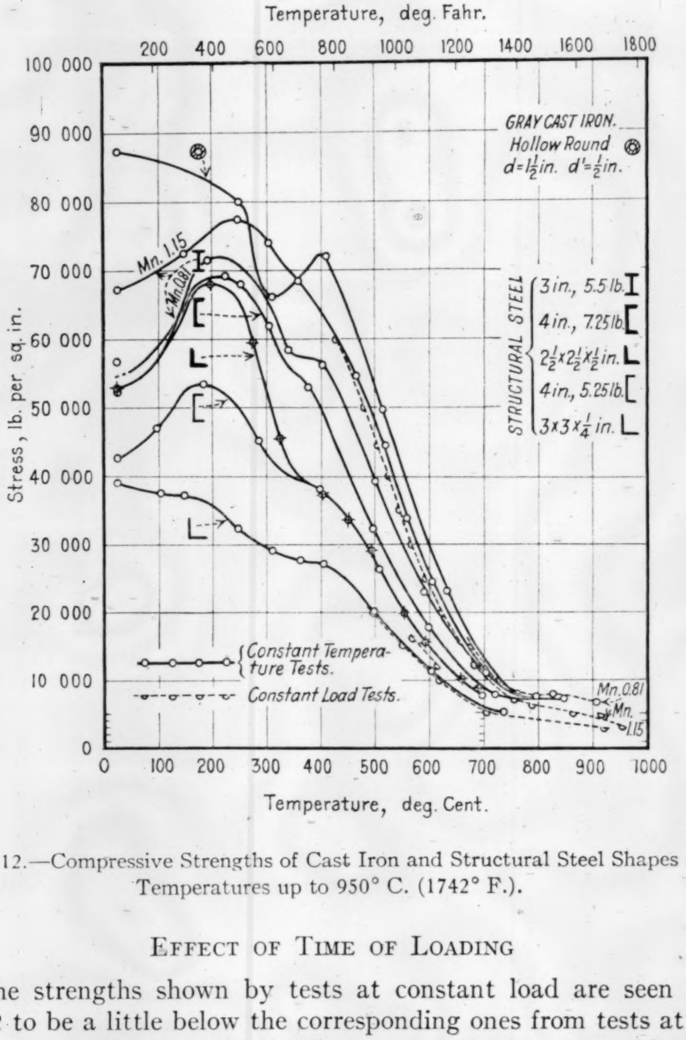

FIG. 12.—Compressive Strengths of Cast Iron and Structural Steel Shapes at Temperatures up to 950° C. (1742° F.).

EFFECT OF TIME OF LOADING

The strengths shown by tests at constant load are seen from Fig. 12 to be a little below the corresponding ones from tests at constant temperature, due without doubt to the longer time of loading. The tests at constant temperature required about 20 minutes and the loading near the ultimate only a few minutes. In the tests at constant load the load was applied initially and the temperature raised at the rate of 2 to 3° C. per minute until failure occurred, which would give a longer time in the temperature region near failure than in the load region near failure in tests at constant temperature. A further study was made on the effect of time of loading with the I-beam section (manganese 0.81 per cent) as shown in Fig. 4. At 592° C. (1098° F.) failure in the regular constant-temperature test occurred at 23,200 lb. per sq. in. In another test at temperature of 595° C. (1103° F.) a constant load of 14,000 lb. per sq. in. was maintained for 2 hours 53 minutes, producing a deformation of 6.9 per cent, after which the

TABLE II.—EFFECT OF LENGTH OF SPECIMENS.

Section	Length, in.	Slenderness Ratio, $\frac{l}{r}$	Temperature		Compressive Strength, lb. per sq. in.
			deg. Cent.	deg. Fahr.	
(a) TESTS AT CONSTANT TEMPERATURE.					
4-in. 7.25-lb. channel	10.5	22.8	Room temperature		55 000
4-in. 7.25-lb. channel	2.41	5.2	Room temperature		67 000
4-in. 7.25-lb. channel	10.5	22.8	603	1117	17 500
4-in. 7.25-lb. channel	2.45	5.3	603	1117	19 750
$2\frac{1}{2}$ by $2\frac{1}{2}$ by $\frac{1}{2}$-in. angle	10.5	22.3	588	1090	15 800
$2\frac{1}{2}$ by $2\frac{1}{2}$ by $\frac{1}{2}$-in. angle	5.25	13.1	588	1090	20 000
4-in. 5.25-lb. channel	10.5	23.4	Room temperature		43 000
4-in. 5.25-lb. channel	1.83	4.1	Room temperature		60 000
4-in. 5.25-lb. channel	10.5	23.4	613	1135	13 400
4-in. 5.25-lb. channel	2.53	5.6	613	1135	17 500
4-in. 5.25-lb. channel	10.5	23.4	588	1090	16 000
4-in. 5.25-lb. channel	5.32	11.8	588	1090	17 300
(b) TESTS AT CONSTANT LOAD.					
Average light angles and channels[a]	10.5	20.6	635	1175	9 800
Plate and angle column No. 23[b]	152.0	111.8	635	1175	8 900
Average light angles and channels[a]	10.5	20.6	609	1128	11 500
Plate and channel column No. 24[b]	152.0	64.7	609	1128	12 650
Average light angles and channels[a]	10.5	20.6	610	1130	11 500
Plate and Z-bar columns No. 25[b]	152.0	81.7	610	1130	11 250
Average light angles and channels[a]	10.5	20.6	598	1108	12 500
Latticed angle column No. 26[b]	152.0	40.7	598	1108	14 500
$1\frac{1}{2}$-in. hollow cast iron[a]	9.2	23.4	734	1353	8 700
7-in. hollow cast iron, average for columns 27, 46, 62 and 63[b]	152.0	68.2	734	1353	6 500

[a] From present series of tests.

[b] From "Fire Tests of Building Columns," Underwriters' Laboratories edition, p. 122; also *Technologic Paper No. 184*, U. S. Bureau of Standards, p. 136.

load was increased to failure at 23,000 lb. per sq. in. At the same temperature a stress of 12,000 lb. per sq. in. was maintained for 4 hours and produced a deformation of 7.5 per cent without actual failure.

At 702° C. (1296° F.) failure in the regular constant temperature test corresponds to a stress of 10,800 lb. per sq. in. as obtained from the strength-temperature curve for this section. At or near the same temperature a stress of 9000 lb. per sq. in. was sustained for only 16 minutes, and 7000 lb. per sq. in. for 38 minutes. A stress of 5000 lb. per sq. in. was maintained for 3 hours 7 minutes, which caused a deformation of 8.6 per cent without producing failure. However, even the loads which did not actually cause failure produced deformations that probably would have caused failure in building columns of ordinary length, for which the limit appears to be from 0.5 to 1.5 per cent for failure under constant load in this temperature region, when tested as individual members.[1]

EFFECT OF LENGTH

Comparisons of compressive strengths are given in Table II on tests at constant temperature for full length (10.5 in.), half length and one-fourth length specimens in the present series. For tests at constant load, the comparisons are on results with the $10\frac{1}{2}$-in. length in the present series and columns 12 ft. 8 in. long that in the column tests previously referred to exhibited a fair degree of temperature uniformity near failure and were without protections that carried any appreciable portions of the load. In tests at constant temperature the increase in strength for the one-fourth length specimen averages 31 per cent for tests at room temperature and 22 per cent for higher temperatures. For the half-length specimens, the average increase over the full-length specimens is 17 per cent at 588° C. (1080° F.).

The average for the four comparisons of structural steel at constant load gives nearly the same value for $10\frac{1}{2}$-in. specimens of light angles and channels in the present series as for the full-size columns, at average temperature of 613° C. (1135° F.). The values for the $10\frac{1}{2}$-in. length are in all cases taken from the strength-temperature curves in Fig. 12. The short cast-iron specimen gave a strength at 734° C. (1353° F.), 34 per cent above that for four columns failing under working load at this average temperature.

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[1] See "Fire Tests of Building Columns," Underwriters' Laboratories edition, pp. 324–344; **also,** *Technologic Paper No. 184,* U. S. Bureau of Standards, pp. 310–330.

DISCUSSION AND SUMMARY

It is apparent that the increase in compressive strength given by most of the sections in the 250° C. (482° F.) temperature region is not a quantity usable in design, particularly as there is decrease in proportional limit and yield point values. Stresses in structural steel from 20,000 to 10,000 lb. per sq. in. correspond to failure temperatures from 500 to 715° C. (932 to 1319° F.) as tested at constant temperature and a few degrees lower at each limit as tested at constant load. The deformations at failure were, however, much larger than would be developed in building members before failure. Maintaining loads below the ultimate for a few hours caused deformations greater than those under which full-size building members would fail. It appears that the strength that can be developed at a given temperature in a member such as a building column exposed to fire lies between the yield point and the compressive strength from the constant temperature tests.

The comparisons on strength under constant load given in Table II pertain to the less stable of the short specimens tested and symmetrical columns of larger section, and apparently the difference in section was sufficient to offset the length effects. For sections of comparable symmetry and thickness of metal, failure of the column would take place at a point intermediate between the yield point and compressive strength of the short specimens. Beams would fail at lower loads for a given temperature or at lower temperatures for a given load, since the deformation that the flanges can withstand before failure is less.

For all conditions where freedom from objectionable permanent deformation is to be assured, protections for the metal are required for given load and fire conditions that will prevent the yield point, as defined in these tests, from being exceeded.

THE CAUSE AND PREVENTION OF EMBRITTLEMENT OF BOILER PLATE[1]

By S. W. Parr[2] and F. G. Straub[3]

Synopsis

Three types of cracks are recognized: first, those due to direct corrosion of the metal; second, those due to fatigue; and third, those which are caused by caustic solutions. Each is found to be distinctive in type, which makes it possible to recognize the embrittlement cracks when met with. A study of the areas throughout the United States where embrittlement occurs has been made, but instances of embrittlement are noted which are the result of boiler water treatment. Hence, it is more important to know the condition of the water rather than the locality from which it comes, as an index of potential danger.

A method of procedure was devised whereby the embrittlement effect could be produced at will, thus making it possible to study both the conditions under which it occurs and remedies for its prevention. The results indicate that two conditions must be present simultaneously to produce the embrittlement effect: first, the stressing of the metal above the yield point; and second, the concentration of sodium hydroxide to a point which is in excess of 350 g. per liter. Parallel tests omitting the sodium hydroxide show no effect. Tests upon various types of metal from the purest obtainable to those with a high percentage of impurities show that impurities do not modify the conditions.

Inhibition of embrittlement, so far as the experiments have gone, indicate that this might be accomplished by the elimination of localized stresses, which is assumed to be impossible. The modification or control of the chemical condition of the water has been found to be effective. Data on actual plants in use, covering a period of ten years, are consistent with the artificial embrittlement in that by maintaining a ratio of sodium sulfate to sodium hydroxide in excess of 2, no embrittlement is found to occur. Free sodium carbonate in the water is in itself not active in producing embrittlement, but contains a potential danger in that it gradually is hydrolized into the caustic form.

Photographs illustrating the cases of actual embrittlement in widely distributed regions, from Buffalo to Southern California, and from Michigan to Texas, show the wide distribution of the difficulty. Photomicrographs of both the natural and the artificially produced cracks furnish a method of identification as well as illustrate their special characteristics.

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[1] Presented by permission of the Director of the Engineering Experiment Station of the University of Illinois, Bulletin of the Station No. 155 under the same title will be ready for distribution in September, 1926.

[2] Professor of Applied Chemistry, University of Illinois, Urbana, Ill.

[3] Special Research Assistant, University of Illinois Engineering Experiment Station, Urbana, Ill.

INTRODUCTION

Within recent years, a phenomenon of more or less frequent occurrence has become recognized, to which the term "the embrittlement of boiler plate" has been applied. Because of the relatively infrequent occurrence of this difficulty and the more or less obscure causes which bring it about, the fact of embrittlement has been largely in question, and both the evidence of its presence and the conditions which promote it are matters of importance at the present time. This is all the more accentuated by reason of the fact that seemingly within recent years an increased number of boiler failures chargeable to embrittlement are in evidence.

It should be said at the outset that these studies were undertaken by the Engineering Experiment Station of the University of Illinois as a continuance of former studies, under conditions which would preclude any possibility of the work being done for any manufacturing or industrial enterprise, but solely for the purpose of developing information of general interest to all, whether concerned in the production of power or the fabrication of boilers. Acknowledgment is here extended with very great appreciation to all of these sources of information. Under these strictly neutral conditions a comprehensive mass of data has been put at our disposal, and wherever reference to the source of such data is given it is distinctly with the permission of the interests involved, and where sources of reference are withheld it is with the understanding that such procedure is preferred. In brief, an attempt has been made to follow methods both in the assembling of information and in the acquiring of experimental data which would avoid any question as to the authenticity of the facts or the disinterested character of the experimental results obtained.

DEVELOPMENT OF THE EMBRITTLEMENT IDEA

It will not be necessary at this point to review the historical development of the study of the phenomenon of embrittlement. A bibliographic compilation elsewhere will be sufficient to cover that feature. Reference only will here be made to the fact that in the early stages of experience along this line, connection seems to have been made with failures of fabricated metal which was used in processes where caustic solutions were involved, as in the manufacture or use of sodium hydroxide, and under these conditions the term "caustic embrittlement" was employed. It was soon evident that a certain relation existed between embrittlement resulting from the use of actual caustic solutions and boiler waters where embrittlement

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It should be said at the outset that these studies were undertaken by the Engineering Experiment Station of the University of Illinois as a continuance of former studies, under conditions which would preclude any possibility of the work being done for any manufacturing or industrial enterprise, but solely for the purpose of developing information of general interest to all, whether concerned in the production of power or the fabrication of boilers. Acknowledgment is here extended with very great appreciation to all of these sources of information. Under these strictly neutral conditions a comprehensive mass of data has been put at our disposal, and wherever reference to the source of such data is given it is distinctly with the permission of the interests involved, and where sources of reference are withheld it is with the understanding that such procedure is preferred. In brief, an attempt has been made to follow methods both in the assembling of information and in the acquiring of experimental data which would avoid any question as to the authenticity of the facts or the disinterested character of the experimental results obtained.

DEVELOPMENT OF THE EMBRITTLEMENT IDEA

It will not be necessary at this point to review the historical development of the study of the phenomenon of embrittlement. A bibliographic compilation elsewhere will be sufficient to cover that feature. Reference only will here be made to the fact that in the early stages of experience along this line, connection seems to have been made with failures of fabricated metal which was used in processes where caustic solutions were involved, as in the manufacture or use of sodium hydroxide, and under these conditions the term "caustic embrittlement" was employed. It was soon evident that a certain relation existed between embrittlement resulting from the use of actual caustic solutions and boiler waters where embrittlement seemed to occur, by reason of the fact that in the case of such boilers the water used was also shown to be caustic. Hence the general term "caustic embrittlement" which is often applied to this phenomenon might seemed to be justified, but in the present discussion only the general term "embrittlement" is employed. In this connection reference is made to a previous study published by the Engineering Experiment Station of the University of Illinois.[1] In this study an attempt was made to establish the connection between caustic solutions and the embrittling action. Also very brief references (pages 13 and 46 of that bulletin) were made to possible methods for prevention of the difficulty. It was appreciated at the time of the publication of *Bulletin No. 94* that information on both the cause and prevention of embrittlement was very meager, and this fact has been a large factor in again reviving the studies on that topic. The seemingly increased number of boiler failures should also be cited as an added reason for again taking up the subject.

It was determined at the outset that it seemed desirable to make a critical survey of the situation with a view to establishing beyond any question the facts *pro* or *con* as to the actual existence of such a thing as embrittlement. There has come somewhat persistently into the discussion of the topic references both to embrittlement as an actual fact and discussions making use of the expression "the embrittlement myth," implying a question as to the reality of embrittlement. Hence the initial phase of these studies was directed towards proof of either the existence or the absence of the phenomenon.

In order to study the matter of embrittlement, therefore, it was deemed essential at the outset to develop some method of recognition which would be reliable without question, and distinctly differentiate such phenomena from other similar or related conditions. This led directly to a study of the cracking of rolled or fabricated plate. The studies along this line have revealed the fact that three types of cracks may occur as follows:

1. Corrosion cracks;
2. Fatigue cracks; and
3. Embrittlement cracks.

Each of these three types is distinctive and can be definitely differentiated from the others by micrographic analysis. Their characteristics may be briefly described as follows:

Corrosion Cracks.—As the name implies, such cracks are due to direct corrosion of the metal. It is well known that where parts of the metal differ in composition or physical properties from other parts

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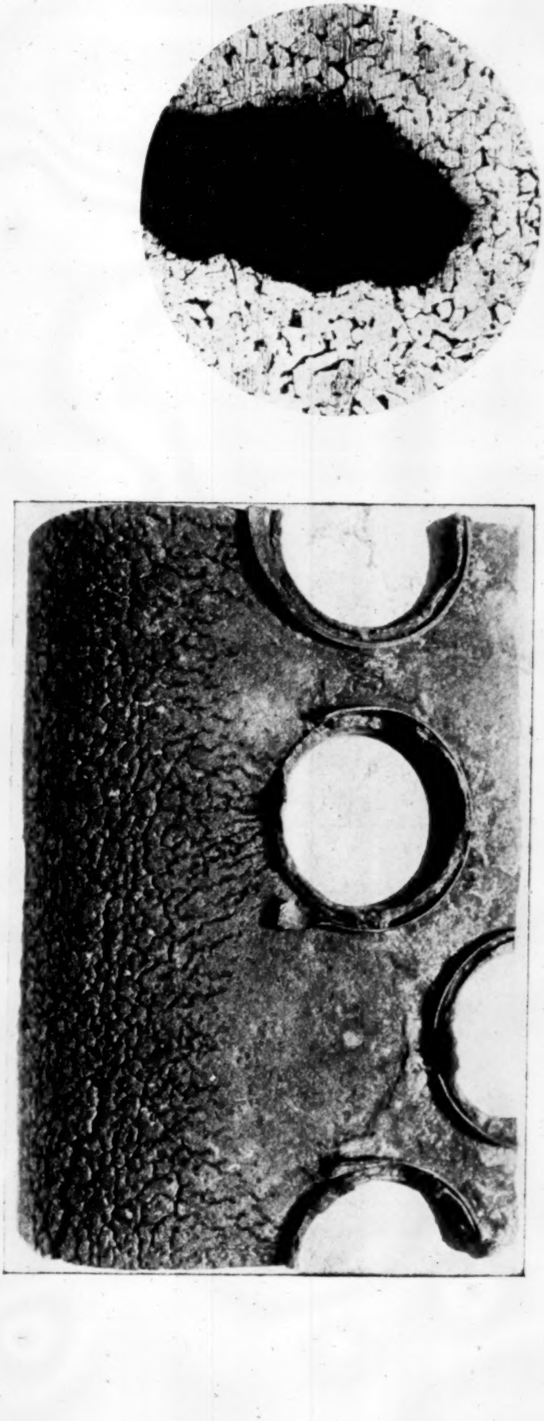
[1] *Bulletin No. 94*, "The Embrittling Action of Sodium Hydroxide on Soft Steel," January 1, 1917.



(b) (×50)

(a)

FIG. 1.—Corrosion Cracking.

due to strains, density or impurities, there exists a difference of electric potential by reason of the fact that one portion of the metal is electropositive to another. This electric potential sets up a galvanic circuit and in the presence of an electrolyte there is started a solution action on the positive side and a corrosion area is thereby developed. These corrosion areas or cracks in the case of metal under strain will follow the lines of stress as we would naturally expect. Also, since the electrolyte for producing the solvent action is accentuated or occurs in the presence of that type of ionization wherein the hydrogen ions predominate, we would expect to find such examples of corrosion in the presence of such electrolytes as nitrates, chlorides, or sulfates, and such corrosion phenomena would be checked or inhibited by the presence of hydroxyl ions or that condition where an alkaline state exists. This in itself will explain why we would not expect to find corrosion cracks in boiler waters which are alkaline in character. A study of chemical corrosion as above described does not necessarily require a micrographic analysis of the structure of the corroded surface and the accompanying illustrations (Fig. 1) include both the unmagnified and the magnified surfaces of cracks of this character. One specific feature should be noted in the micrograph, namely, the direction of the cracks follows the lines of stress, and this without regard to grain areas, that is, they proceed across the grains and disregard the grain boundaries in their path of development.

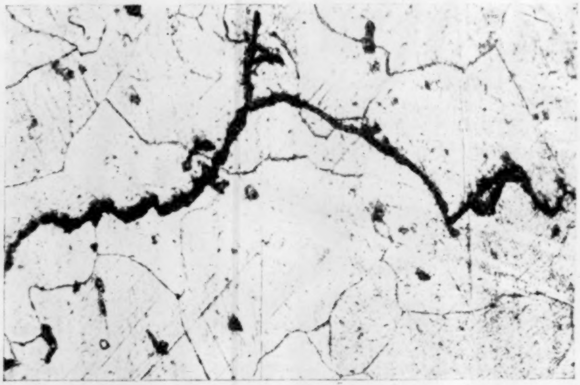
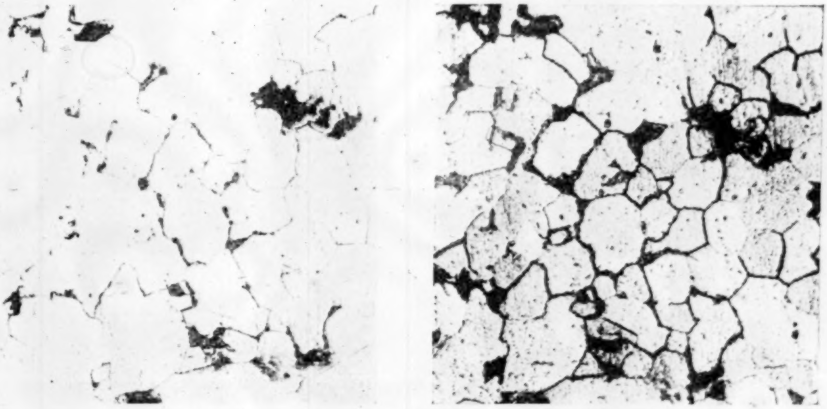


Fig 2.—Micrograph of Fatigue Cracks in Armco Iron (×350). Etched with 2-per-cent Nital Solution.

Fatigue Cracks.—The development of cracks due to stress alone furnishes interesting illustrative material in the study of metal cracks. Because of the abundance of material available of this type from the investigations of the fatigue of metals laboratory, an illustration is here introduced (Fig. 2) because of its bearing on the general topic of cracking of plate. It seems that in metals placed under reverse strains, as failure is approached, cracks develop which upon micrographic study reveal a positive characteristic which serves as a ready method of identification. By examination of a few type specimens it is obvious that the cracks which have been started as a result of fatigue do not respect the fact of grain structure in the metal, but follow a course quite independent of grain boundaries and are hence described as cross-granular in character as opposed to inter-granular in their directional development.



(*a*) Unetched (×250) (*b*) Etched (×250)

FIG. 3.—Micrographs of Embrittlement Cracks from Boilers.

Embrittlement Cracks.—We are now able to differentiate those cracks which accompany embrittlement from either of the above types for the reason that a micrographic study shows one distinctive characteristic which in all cases is found to accompany this type. By reference to the micrographs of embrittlement cracks, especially when the surface is etched in such manner as to bring out clearly the grain boundaries, it is very evident that without exception these embrittlement cracks follow grain boundaries. Two typical micrographs illustrating the point are shown in Fig. 3.

With this very definite method of identifying any of the cracks that may occur in connection with boiler plate, it has been possible to make an extended survey of the situation geographically, as well as from the standpoint of boiler fabrication and of boiler-water treatment.

It would be impossible within a reasonable space to give in detail the evidence of embrittlement which has thus been assembled in connection with these studies. A few typical samples only are made use of. In general the cases of embrittlement may be discussed under two headings:

1. Regional areas where a specific type of natural water exists;
2. Treated waters resulting from the direct application of a chemical resulting in embrittlement.

Under the first topic it is clearly evident that certain regional areas may be defined in which embrittlement is more evident than in other regions where embrittlement does not exist. The waters of these regions are characterized by the fact of an almost complete absence of sulfates in the water, but this phenomenon accompanies another very marked characteristic and the one which is primarily responsible for the embrittling action, namely, the presence of free sodium bicarbonate. It will thus be seen that these waters have only temporary hardness due to the presence of bicarbonates of lime, magnesium and iron, and that there is a substantial absence of sulfates of these elements. Areas of this type, without attempting to circumscribe such areas, may be in a general way located as shown on the map in Fig. 4. This does not necessarily mean that in the collection of data of this sort we pretend to cover all of the localities where

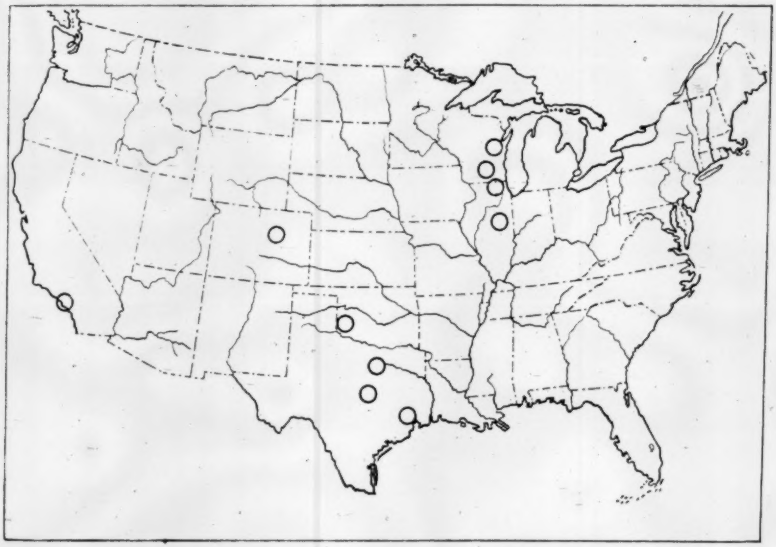


FIG. 4.—Areas in Which Boilers Using Well Waters Have Been Embrittled.

waters of this type are met with. As a matter of fact, since the transition from shallow wells to deeper sources of supplies has progressed, waters from isolated wells without regard to any defined areas may be met with in almost any region.

In the second case, as will be seen later, the causes of embrittlement which can be traced to water treatment are comparatively few in number, but the possibilities of such distress if even remotely present should be thoroughly understood and the knowledge of how to avoid the difficulty should be quite as eagerly sought by water-treating establishments as by the users of such water-treating formulas or apparatus.

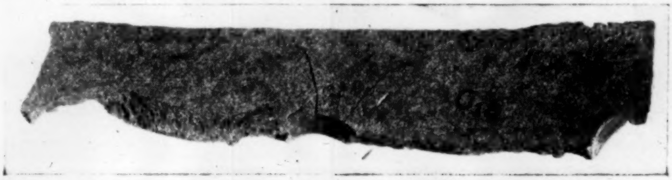
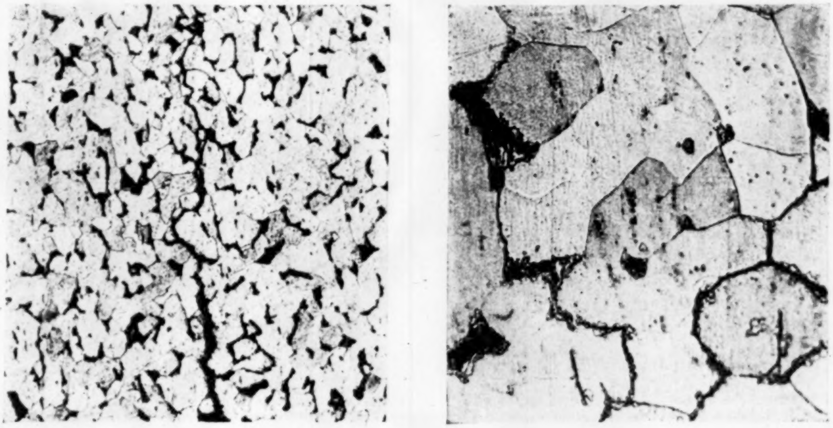


FIG. 5.—Section of Cracked Plate from Boiler Using Treated Water.



(*a*) (×100)

(*b*) (×500)

FIG. 6.—Micrographs of Cracks in Embrittled Plates. Etched with 2-per-cent Nital Solution.

An illustration is given in Fig. 5, but it should be emphasized that it represents only one case of this type.

Two other phases of the problem should receive brief consideration.

The confining of the distress to any particular make or type of boiler is wholly without confirmatory evidence. That is to say, we have found instances of embrittlement in boilers of different makes and designs in sufficient number to warrant the statement that the embrittling effect is not confined to any particular make or type of boiler, all of the standard makes of both fire and water-tube boilers having been represented in the investigation.

Not a little controversy has centered in the question as to whether the embrittling action may not be due to faulty composition of the iron. This question seems to have been decided in our studies to the effect that the embrittling effect occurs without regard to impurities or composition of the plate and that any defence of the embrittling distress attempting to charge it against faulty iron is without basis in fact. By this we mean that embrittlement is not confined to faulty or dirty iron, but quite as consistently occurs in iron of the best grade as otherwise. The micrographs in Fig. 6 will serve to illustrate these facts.

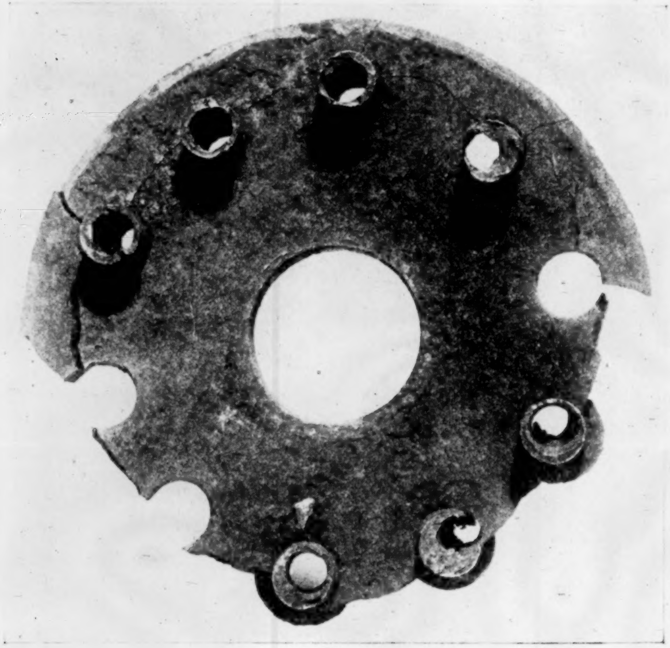


FIG. 7.—Embrittled Blow-Off Flange.

SUMMARY OF EMBRITTLEMENT CHARACTERISTICS

The salient features which are characteristic of embrittlement may be more fully understood upon a brief survey of a few typical illustrations taken from the numerous cases which have come to our attention.

Fig. 7, for example, is a blow-off flange and it may be inferred, from the frequency with which cases of this sort are met, that the difficulty may be accentuated by the vibratory effect produced by the blowing-off process.

Fig. 8 is the outside of a drum from which twenty-six rivet heads were knocked off by sharp blows from a hammer. Figs. 3 and 6 show micrographic studies of embrittlement cracks in these same or corresponding plates which have failed in service.

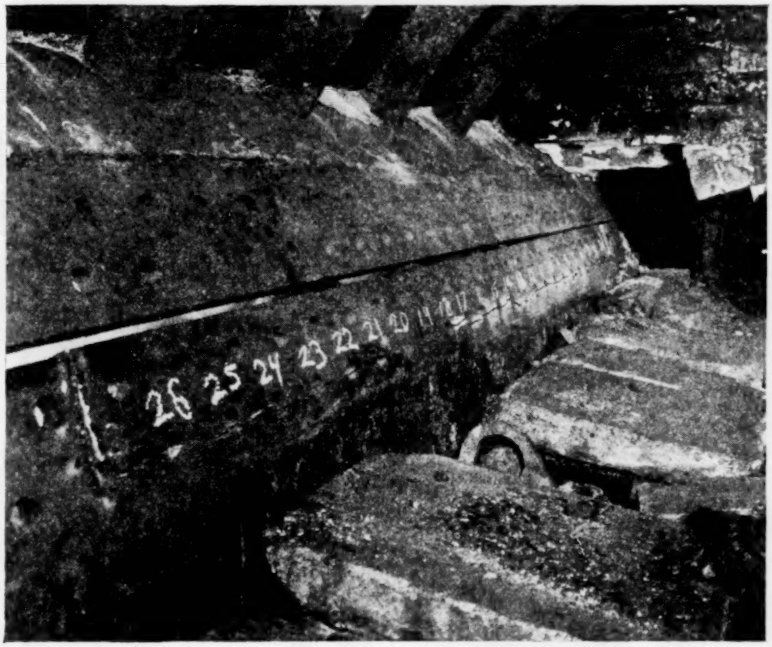


FIG. 8.—Outside of Embrittled Drum in a 500-h-p. Boiler.

The characteristics of these cracks may be summarized as follows:

1. The cracks do not follow what is generally considered the line of maximum stress;

2. They start on what is termed the dry side of the plate;

3. They run, in general, from one rivet hole to another, though they often run past each other, leaving islands of plate;

4. They are irregular in direction;

5. They never extend into the body of the plate beyond the lap of the seam;

6. There is no elongation of the plate;

7. Where extreme action has occurred, rivet heads crack off or are easily dislodged by a slight blow of the hammer.

A summary of conditions relating to the location of the cracks in the boiler may be given as follows:

1. The cracks always occur below the practical water level;
2. All the cracks are in seams under tension;
3. They occur at places where the highest localized stresses might be assumed to occur;

TABLE I.—CHEMICAL ANALYSIS OF PLATES FROM EMBRITTLED BOILERS.

Location of Boiler	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent
Bloomington, Ill	0.17	0.26	0.015	0.046
Hartland, Ill	0.22	0.39	0.017	0.024
Champaign, Ill.	0.20	0.50	0.005	0.024
Los Angeles, Calif	0.22	0.54	0.013	0.036
Houston, Texas	0.26	0.34	0.014	0.025
Railway	0.14	0.37	0.018	0.018

TABLE II.—ANALYSIS IN GRAINS PER U. S. GALLON OF WELL WATERS WHICH HAVE BEEN USED IN EMBRITTLED BOILERS.

	Bloomington, Ill.	Urbana, and Champaign, Ill.	DeKalb, Ill.	McHenry County, Ill.	Watseka, Ill.	Paxton, Ill.	Sycamore, Ill.	Los Angeles, Calif.	Dallas, Texas	Denver, Colo.	Houston, Texas
Calcium Carbonate	9.27	9.75	8.23	6.27	5.55	9.97	9.22	3.55	1.47	0.60	2.58
Magnesium Carbonate	6.82	6.34	4.72	5.18	2.99	6.64	6.26	0.38	1.01	0.26	0.00
Sodium Carbonate	3.21	4.52	3.93	3.12	8.82	3.96	1.99	7.71	21.70	5.96	7.1
Sodium Sulfate	0.00	0.10	0.08	0.00	0.83	2.36	0.16	5.72	11.92	1.49	0.0
Sodium Chloride	3.85	0.47	0.10	0.39	0.54	0.25	0.24	5.10	4.21	0.70	12.1
Iron Oxide and Aluminum Oxide	0.12	0.10	0.07	0.53	0.09	0.03	0.17	....	....	0.05	0.0
Silica	0.58	0.82	0.40	0.23	0.31	1.82	1.18	0.95	2.51	0.63	....
Total Solids	26.35	23.17	17.92	16.03	20.04	27.80	19.45	....	48.50	9.69	....

4. The cracks occur in plates having practically perfect chemical composition and physical properties as well as in plates of inferior make.

Table I gives the chemical analysis of boiler plate in which a few typical cases of embrittlement have occurred. It is of interest to compare these analyses with those of standard samples of high-grade material furnished to us direct from the manufacturers for testing under a later topic, that of "Laboratory Reproduction of Embrittlement."

A summary of the chemical conditions which characterize the water used in boilers where embrittlement has occurred is as follows:

1. Sodium carbonate is the one substance which is always present in the feed water;

2. Sulfate hardness is usually absent or of a low ratio with respect to the sodium carbonate present. Sodium sulfate is similarly lower in amount than the sodium carbonate;

3. Boilers encountering this trouble use waters having the characteristics noted under 1 and 2, and as a consequence of chemical reaction within the boiler develop a caustic condition with the sodium hydroxide in material excess over the sodium sulfate present.

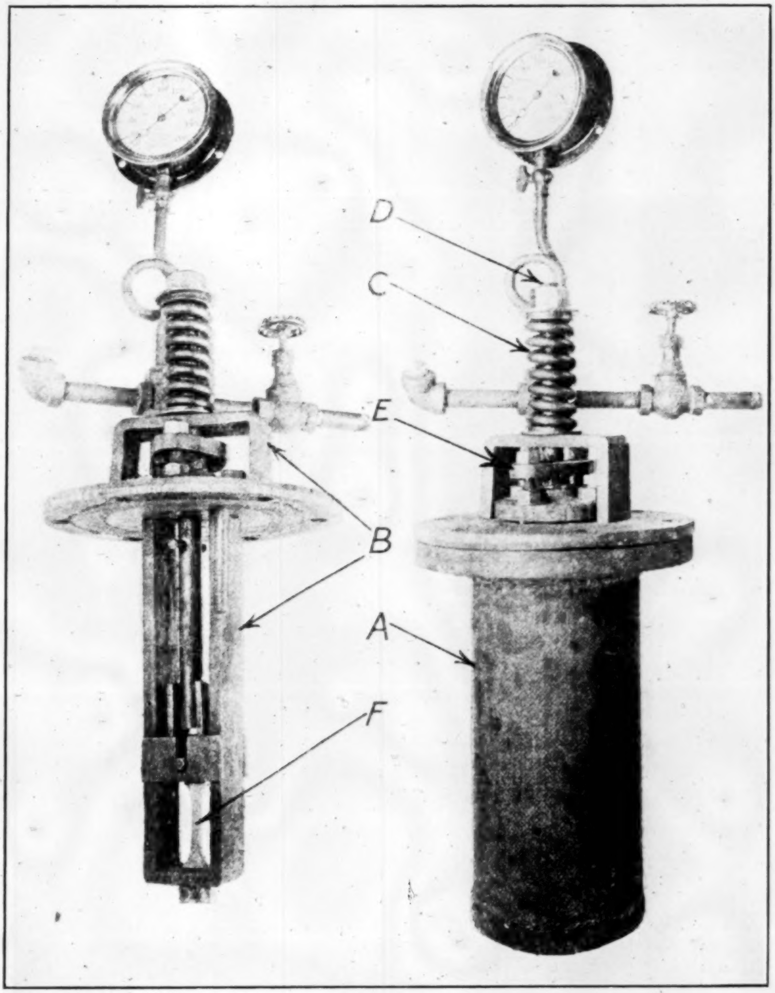


FIG. 9.—Apparatus Used for Embrittlement Tests.

Table II gives the analysis of well waters which have been used in embrittled boilers.

LABORATORY REPRODUCTION OF EMBRITTLEMENT

It will be at once evident that if a method could be devised for reproducing the feature of embrittlement at will, a ready means would be available for determining both the cause of and the remedy for the difficulty. Intercrystallin cracking of mild steel is an abnormal type of failure; consequently if mild steel can be made to crack under conditions which may be controlled or modified at will and under conditions which parallel those of actual boiler operations, both the cause and the remedy may be in a fair way of solution.

TABLE III.—CHEMICAL ANALYSIS OF METALS TESTED.

Reference	Description	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Molybdenum, per cent
F. S.	Flange Steel	0.18	0.45	0.012	0.027	....	....	....
Armco	Armco Iron	0.023	0.017	0.003	0.010	0.01	....	....
M. I.	Magnetic Iron	0.04	0.020	0.004	0.007	0.01	....	....
C. R.	Cold Rolled	0.30	0.42	0.015	0.044	0.04	....	....
1112	S. A. E. 1112	0.15	0.76	0.215	0.126	0.01	....	....
2312	S. A. E. 2312[a], [b]	0.115	0.45	0.018	0.025	0.20	3.5	....
2212	Special[a], [b]	0.13	0.45	0.018	0.020	0.26	1.57	0.25
2330	S. A. E. 2330[b]	0.30	0.478	0.023	0.015	0.05	3.42	....

[a] Furnished by Central Steel Co., Massillon, Ohio.
[b] Cut to $\frac{1}{4}$ in. flat from $1\frac{1}{4}$ in. round.

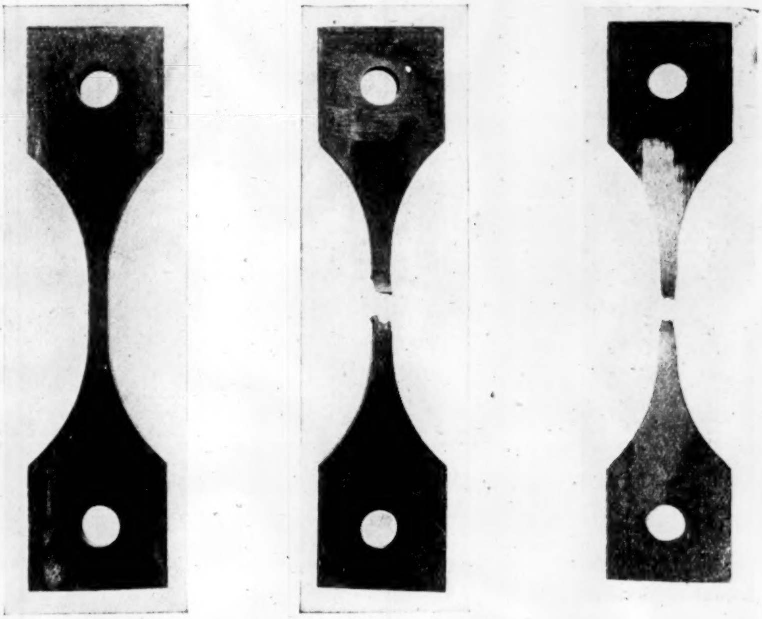
TABLE IV.—TENSION TESTS OF MATERIALS USED.

Reference	Description	Heat Treatment[a]	Elastic Limit, lb. per sq. in.	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Reduction of Area, per cent
F. S.	Flange Steel	As received	32 600	35 200	60 400	61
Armco	Armco Iron	Annealed 950° C	32 000	40 600	52 600	70
M. I.	Magnetic Iron	Annealed 950° C	25 000	30 000	50 700	72
C. R.	Cold Rolled	As received	70 000	84 000	85 500	55
C. R. A.	Cold Rolled	Annealed 850° C	.....	36 400	64 700	55
1112	S. A. E. 1112	Annealed 900° C	47 500	49 000	74 000	56
2312	S. A. E. 2312	Annealed 900° C	37 000	40 000	74 800	75
2212	Special	Annealed 900° C	35 000	36 800	75 000	75
2330	S. A. E. 2330	Annealed 850° C	41 000	49 200	77 300	57

[a] Annealed specimens heated to temperature indicated for 30 minutes and furnace cooled.

The value of direct accessibility of information obtained in this way may be well illustrated by the only other means, namely, a long-time experiment in connection with boilers in actual operation. Since the embrittling effect is insidious and in many cases very slow in developing, it will at once be understood why this method of experimentation is almost hopeless because of the time element involved. However, it should be said that one such experiment extending over the time intervening since publication of the former bulletin, namely, ten years, has been in progress, and all of the results obtained in that connection are consistent with and indeed are substantiated by the results of these short-time laboratory tests.

The apparatus finally used for reproduction of embrittlement is shown in Fig. 9. It consisted of a welded steel container *A* which held the solution and the tension producing equipment *B*. The tension was produced and maintained by means of the spring *C* and the plunger *D*. The gland *E* allowed the plunger to enter the container steam tight. The specimen is shown at *F*. Part *B* fitted into *A* and was secured by bolts (not shown). The proper temperature and pressure were maintained by means of an electric furnace.



(*a*) Test Specimen. (*b*) Embrittled Specimen. (*c*) Tension Test Specimen.

FIG. 10.—Test Specimens.

The materials used for the embrittling tests were all mild steels. Table III gives the chemical analyses and reference numbers of the different metals tested. Table IV gives the physical properties of the metals.

The materials to be tested were cut to the shape shown in Fig. 10. Care was taken in machining the specimens to have the holes aligned so as to properly distribute the stress. The specimens were finished by polishing the small section with No. 1 polishing emery paper. The direction of polish was parallel to the length of the specimen.

The specimen to be tested was measured with a micrometer and the area calculated. The load necessary to give the required stress was calculated and the compression of the standardized spring to give this load determined. The specimen was set in the yoke and pinned to the plunger which was tightened by screwing down the nut on the upper end. When the spring was compressed to the right length the gland was tightened and the upper part of the apparatus placed on the container in which the desired solution had been previously placed. After the parts had been tightly bolted together they were placed in an electric furnace and heated until the desired pressure was obtained. A record of the pressure, temperature, and spring length was taken at regular intervals. When embrittlement had progressed sufficiently to break the specimen, the spring forced the plunger up, thus indicating that the specimen had broken. The release of the spring was in all cases found to be instantaneous and free from any indications of slow yielding.

Table V.—Effect on Flange Steel of Stress and Concentration of Solution.

Solution, g. NaOH per liter	Stress, lb. per sq. in	Container	Time		Gage Pressure, lb. per sq. in.
			Break	No Break	
415	30 000	No. 1	.....	22 days	65
400	31 600	No. 4	.....	18 days	65
400	33 000	No. 4	.....	16 days	70
415	34 400	No. 1	.....	27 days	90
400	35 400	No. 1	$1\frac{1}{2}$ days	.....	60
400	39 200	No. 4	2 days	.....	55
400	44 300	No. 4	30 hours	.....	50
400	46 000	No. 2	$1\frac{1}{2}$ days	.....	45
400	50 500	No. 4	27 hours	.....	50
40	50 000	No. 1	.....	17 days	55
200	47 000	No. 3	.....	16 days	40
210	47 600	No. 3	.....	21 days	65
345	46 000	No. 4	$14\frac{1}{2}$ days	.....	65
405	50 000	No. 4	$4\frac{1}{2}$ days	.....	50
400	39 200	No. 4	2 days	.....	55
455	52 000	No. 3	$2\frac{3}{4}$ days	.....	95
575	46 700	No. 3	$8\frac{1}{2}$ days	.....	50

Table VI.—Effect on Flange Steel of Solutions of Salts other than Sodium Hydroxide.

Salt Used	Concentration, per liter	Stress, lb. per sq. in.	Container	Time No Break, days	Gage Pressure. lb. per sq. in.
None	Distilled Water	48 600	No. 1	28	50
Na_2CO_3	500	50 000	No. 3	34	70
Na_2SO_4	500	45 000	No. 4	30	70

The average area of the test specimens was 0.05 sq. in. The accuracy of the estimated load on the specimen was within 1000 lb.

TABLE VII.—EFFECT ON FLANGE STEEL OF PREVIOUS HEAT TREATMENT, CHEMICAL ACTION AND PRESSURE.

Solution, g. NaOH per liter	Stress, lb. per sq. in.	Container	Time		Gage Pressure, lb. per sq. in.	Treatment
			Break	No Break		
400	39 200	No. 4	2 days	....	55	No treatment
400	50 000	No. 2	2 days	....	55	Annealed 1050° C.
418	50 000	No. 2	20 hours	....	55	No treatment
407	50 000	No. 2	20 hours	....	45	Dilute HCl 20 minutes
407	50 000	No. 2	17 hours	....	55	Lower half $PtCl_3$ Solution
400	50 000	No. 2	36 hours	....	55	All $PtCl_3$ Solution
404	50 000	No. 4	....	16 days	0[1]	
400	46 000	No. 2	1½ days	....	45	
390	50 000	No. 2	2 days	....	200	

[1] Open to air.

TABLE VIII.—EFFECT OF COLD WORK.

Specimen Tested	Stress Before Testing	Stress During Test, lb. per sq. in.	Container	Time		Gage Pressure, lb. per sq. in.
				Break	No Break	
F. S.	....	39 200	No. 4	2 days	....	50
F. S.	55 000 lb. per sq. in..............	25 000	No. 2	....	22 days[a]	50
F. S.	Cold worked 20 per cent elongation Continued........................	25 000	No. 1	....	27 days	55
F. S.	....	45 000	No. 1	12 hours	....	60
C. R.	....	25 000	No. 2	....	24 days	85
C. R.	....	38 000	No. 2	....	24 days	85
C. R.	....	45 000	No. 2	17½ days	....	80
C. R.	....	55 000	No. 2	28 hours	....	85
C. R. A.	....	38 000	No. 2	36 hours	....	85

[a] Tested for tensile strength after 22 days in solution. Tensile strength 69 200 lb. per sq. in.; reduction of area 52 per cent.

TABLE IX.—EFFECT OF CHEMICAL COMPOSITION.

Specimen Tested	Solution, g. NaOH per liter	Stress, lb. per sq. in.	Container	Time		Gage Pressure, lb. per sq. in.	Yield Point, lb. per sq. in.
				Break	No Break		
Armco	409	40 200	No. 2	....	14 days	75	40 600
Armco	400	45 000	No. 2	21 hours	....	70	40 600
M. I.	405	35 000	No. 2	23 hours	....	50	30 000
F. S.	418	50 000	No. 2	20 hours	....	60	35 200
1112	409	55 000	No. 2	3½ hours	....	50	49 000
1112	400	40 000	No. 2	15 hours	....	80	49 000
1112	427	35 000	No. 4	....	20 days	80	49 000
1112	427	25 000	No. 4	....	25 days	85	49 000
2312	375	48 000	No. 2	29 hours	....	80	40 000
2312	450	43 000	No. 4	8½ days	....	85	40 000
2212	385	45 000	No. 2	20 hours	....	65	36 800
2330	375	55 000	No. 4	3½ days	....	65	49 200

per sq. in. when the stress was in the range of 40,000 lb. per sq. in. In calculating the stress, allowance was made for the stress added by the steam pressure acting on the plunger.

TEST DATA ON EMBRITTLING

The results of the tests on the various metals embrittled are given in Tables V to IX, inclusive. They indicate that two conditions must be present simultaneously to cause embrittlement of steel: first, the actual stress must be above the yield point of the metal; and second, the concentration of sodium hydroxide must be in excess of 350 g. per liter (350,000 parts per million, 20,000 grains per gallon). The variation of pressure up to 200 lb. per sq. in. seems to have no marked effect on the rate of embrittlement.

When distilled water, sodium-carbonate solution, or sodium-sulfate solution was used in place of sodium hydroxide, the metal was not affected even when the stress was equal to that causing failure when sodium hydroxide was used.

The temperature of the previous complete annealing sample has no marked effect on the rate of embrittlement, neither has the amount of stress once it passes the yield point.

If the metal has been extremely cold-worked previous to testing for the rate of embrittlement, it will not lower the stress necessary to start this effect, but it appears that it will actually require a higher stress than would be required for the original unworked metal.

The change of chemical composition of the metal within the limits set for flange steel has very little effect on the rate of embrittlement. Thus, steels of the following general composition show no variation in behavior in respect to embrittling action.

Carbon	0.023	to	0.30	per cent
Manganese	0.017	"	0.45	"
Phosphorus	0.003	"	0.012	"
Sulfur	0.007	"	0.027	"

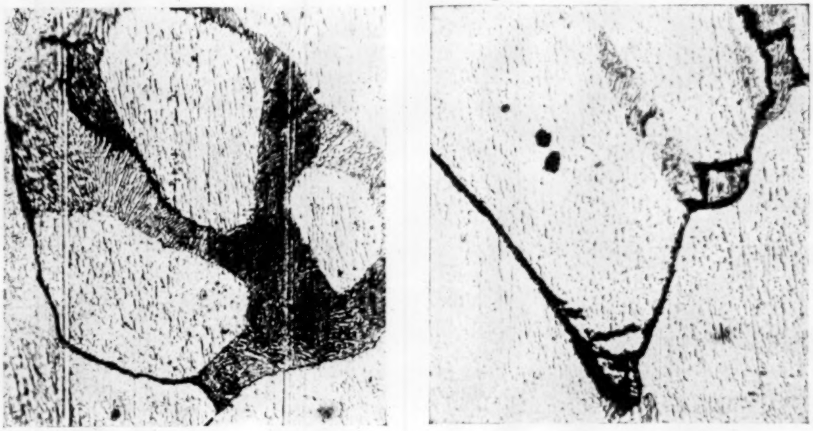
The introduction of 3.5 per cent nickel with the carbon either 0.115 or 0.3 per cent has no effect other than raising the yield point and consequently the initial stress necessary to start embrittlement. A combination of 1.5 per cent nickel with 0.25 per cent molybdenum behaves the same as nickel.

When the sulfur becomes 0.215 per cent and the phosphorus 0.126 per cent, the rate of embrittlement shows a marked acceleration.

The embrittled specimens are not corroded but are covered with a thin, shiny, blue-black, coherent coat of magnetic oxide of iron. They show no marked elongation or reduction of area at the break. Fig. 10 shows an embrittled specimen as compared with a regular tension-test specimen.

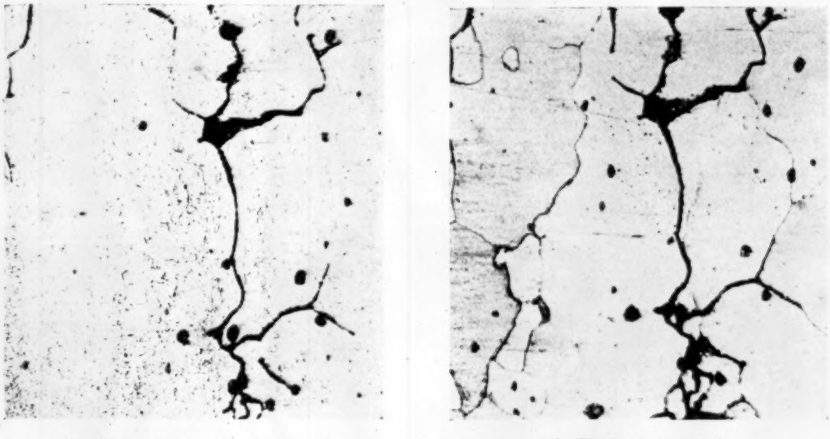
MICRO-EXAMINATION OF EMBRITTLED METALS

Samples of cracked steel were obtained from different instances of this type of boiler distress. These when examined under the miscroscope showed that the cracks were intercrystallin. The manner in which mild steel fails under ordinary stress, either static or fatigue, is almost invariably trans-crystallin. The fact that embrittlement cracks progress between the grains indicates that this is not a normal fracture. Furthermore, the crack progresses without



(a) Etched (×900) (b) Etched (×900)

FIG. 11.—Micrographs of Flange Steel Embrittled in Tests.



(a) Unetched (×250) (b) Etched (×250)

FIG. 12.—Micrographs of Magnetic Iron Embrittled in Tests.

any marked deformation of the grain, another deviation from the general behavior of mild-steel static failures. When examined under higher magnification, the crack is observed to pass between the smaller grains of pearlite which make up the carbon areas.

The specimens embrittled in the laboratory have been examined and found to have a large number of cracks progressing into the strained portion and all of these are intercrystallin without any marked deformation of the grain, as is shown by the micrographs in Figs. 11 and 12.

INHIBITION OF EMBRITTLEMENT

Since embrittlement is the result of the combined action of chemicals and stress on the metal, it appears possible to stop it by at least one of two methods. The stress may be reduced to a point where embrittlement will not occur. This might be possible if the actual concentrated stresses such as at rivet heads, around rivet holes, etc., could be calculated and the boiler constructed so as to keep these stresses low enough. Even this could not be an absolute prevention since some unknown localized stresses might still exist and meet the embrittlement conditions.

TABLE X.—EFFECT OF INCREASING $\frac{Na_2SO_4}{NaOH}$ AND $\frac{Na_2CO_3}{NaOH}$ RATIOS.

Solution, g. NaOH per liter	Ratio		Stress, lb. per sq. in.	Container	Time		Gage Pressure, lb. per sq. in.
	Na_2SO_4 to NaOH	Na_2CO_3 to NaOH			Break	No Break	
455	0	...	50 000	No. 3	$2\frac{3}{4}$ days	....	90
447	0.7	...	50 000	No. 3	$4\frac{1}{2}$ days	....	90
365	1.2	...	40 000	No. 3	$6\frac{1}{2}$ days	....	90
500	1.8	...	40 000	No. 3	$10\frac{1}{2}$ days	....	90
430	2.1	...	40 000	No. 3	....	41 days	100
398	...	0	40 000	No. 1	$2\frac{1}{2}$ days	....	90
415	...	0.3	40 000	No. 1	5 days	....	100
430	...	0.7	40 000	No. 1	11 days	....	100

The removal of the cause of the chemical action, in the absence of which embrittlement would not occur, would be the best procedure; but even this is difficult in some cases by the fact that a change of water is not always possible. A close approach could be made by neutralizing the alkalinity with some chemical, but complete neutralization would be impossible without endangering the boiler.

When it is noticed that embrittlement never has occurred even in carbonate waters when the sulfate content is high, the question is raised as to whether or not the existence of a definite ratio between the sodium sulfate and the sodium hydroxide is sufficient in itself to prevent this cracking. Tests as to the effectiveness of the various ratios of NA_2SO_4 and Na_2CO_3 to NaOH have been run in the laboratory. The results of these tests are given in Table X.

In testing for the inhibiting effect of sodium sulfate and sodium carbonate, it was found that these salts are not very soluble in the hydroxide solution used. When the regular test apparatus was used, the introduction of sulfate up to a ratio of two parts of sulfate to one of hydroxide had no effect on the time of embrittlement. The sulfate was found to be precipitated on the bottom of the container and out of contact with the specimen.

The container was then modified so that the specimen was surrounded by a steel jacket which left a space of about $\frac{1}{32}$ in. around the specimen for the solution to penetrate. A dilute solution was used to start with and concentrated by releasing steam from the container until the proper concentration of the hydroxide was obtained. In this manner conditions approaching those of concentration in the seam of a boiler were obtained. The results tabulated in Table X were obtained using this modification of the testing apparatus. When the containers were opened the inhibiting salts were found to have crystallized on the surface of the test specimen.

The effect of a sulfate-carbonate ratio in plant operation has been studied in one plant for a period of ten years. This plant, the University of Illinois power plant, had been experiencing embrittlement troubles for some time and when in 1915 three new drums had to be replaced after only five years of service the following system of water treatment was inaugurated: The sulfate-carbonate ratio of the feed water is maintained at 2. This is done by neutralizing about 70 per cent of the alkalinity with sulfuric acid. The water is treated in two 40,000-gal. settling tanks. About 110 lb. of lime is added to each tank and after sufficient agitation and settling, the required amount of acid. Analyses of each tank are made daily after lime treatment, and again after the acid is added, to determine total alkalinity. Daily analyses are also made of the water in each boiler for causticity and total alkalinity. After ten years of operation on this treatment the boilers were given a thorough inspection during February, 1926. Test rivets were removed and a close inspection made for signs of leaking or cracks around the rivet holes. At that time the boiler inspector from the Hartford Steam Boiler Inspection and Insurance Co. pronounced the drums to be in perfect condition. The rivets were redriven and the boilers are back in operation.

A system of treatment on a continuous flow principle has been devised and is now in use in several central power plants in the Chicago district. The water is a zeolite water low in the sulfate-carbonate ratio and this ratio is raised by allowing a definite amount of dilute acid to flow into a mixing chamber through which a definite amount of water is being passed. A regular chemical analysis of the boiler water is also maintained at these plants.

A power plant in Champaign had boilers go into service in 1916 using the same type of water as the University of Illinois but without the acid treatment, and in 1925 considerable trouble was experienced due to embrittlement. Fig. 8 shows one of the condemned drums from this plant. These two plants operating for 9 and 10 years, within 20 lb. of the same steam pressure, and upon precisely the same type of water, one sulfate-treated and the other not, serve as a long-time experiment with results that are strictly in accord with the laboratory indications. The boilers using the treated water are in excellent condition while the others have been condemned after only nine years of service.

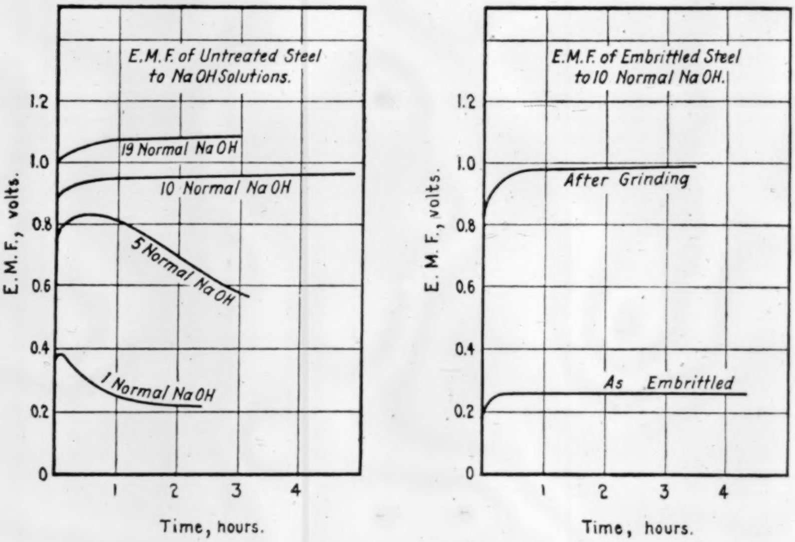


FIG. 13.—E. M. F. of Mild Steel to Sodium Hydroxide Solution.

MECHANISM OF EMBRITTLEMENT

The results that have been obtained justify the opinion which has prevailed in the United States that mild steel is embrittled by sodium hydroxide. They go further in that they show that sodium hydroxide is the only salt in the boiler which will embrittle steel.

Stress and chemical attack now appear to be the predominating factors, neither of which can produce embrittlement in the absence of the other.

The action of sodium hydroxide of various concentrations on mild steel is illustrated in Fig. 13, which gives the e.m.f. of the steel to the solution. These curves show the potential of steel in respect to different concentrations of sodium hydroxide against normal NaOH, HgO and Hg. The measurements were made by the regular compensation method. Fig. 13 shows the e.m.f. of a steel which has been embrittled in the laboratory to 10 normal NaOH. The e.m.f. of the same metal to the same solution after the outer coat has been ground off is also given.

The action of sodium hydroxide is to produce a thin, compact, and coherent coating of magnetic oxide of iron. The reaction with the hydroxylion only is $3\ Fe + 4\ OH = Fe_3O_4 + 4\ H$. If the concentration is kept in the range of 400 g. per liter the action progresses slowly without any noticeable evolution of hydrogen. This coating on the iron makes the iron passive or resistant to further action of the hydroxide. If this coat is cut, removed or penetrated, the fresh metal beneath is as active as the steel was originally. In a solution of 10 normal NaOH (400 g. per liter), the e.m.f. between the fresh metal and the coated metal reaches 0.7 volt in the cold. This potential is sufficient to produce a slow electrochemical action which would favor penetration toward the fresh metal.

As long as the metal is subjected to a stress below the yield point, the chemical action progresses no further than to produce the outer coat of protective oxide. When the stress becomes greater than the yield point of the metal, the outer coat appears to be broken in the strained areas and the chemical attack progresses into the metal to embrittle it. This statement refers to the short-time laboratory tests.

To obtain a clear conception of the possible chemical attack taking place under stress above the yield point of the metal, it becomes necessary to visualize what is taking place within the steel at the yield point. During a static test in which the load is applied at a definite rate, it is found that at the yield point there is a marked increase in the elongation of the metal without any noticeable increase in load. What takes place within mild steel to allow this permanent deformation is at present a matter of conjecture. Undoubtedly it must be due to rearrangement within the metal. The yield point can be said to be a function largely of the crystal strength. When the stress becomes greater than the crystal strength, slip takes place within the crystal. In an aggregate of grains such as there is in mild steel, the slip is stopped partly by interference of adjacent grains and partly by resistance on the slip plane itself. The metal as it is stressed below the yield point is slightly elastic, having an elastic stretch in this range of about 0.1 per cent of the length. As the stress reaches the yield point it becomes sufficient to start slip on the crystal faces. This initial slip is stopped by increase of strength along the slip plane as well as by "end thrust" against adjacent grains. Thus slip in one crystal allows distribution of the stress which in turn increases the stress on the adjacent crystals. At the yield point, it may then be conceivable to think of the realignment of stresses within the metal so as to bring more of the crystal planes and boundaries into opposing the outer stress. After this "reorganization" within the metal there would be a permanent deformation and further external stress would produce additional slip and drawing out of the crystals in the direction of pull. At the time of realignment of stresses within the metal, the stresses at the grain boundaries undoubtedly are of a great order of magnitude. The metal has stretched now to a length more than one per cent of its length. "It is to be assumed that the amorphous cement supposed to exist at the grain boundaries is electropositive to the crystal metal, and therefore has a high internal energy content. This would account for its greater susceptibility to chemical attack."[1] There must also be a storing up of energy when the grain boundaries are so highly stressed, which in turn would make them still more chemically active than they were before stressing.

Once the penetration has started, the chemical attack follows the more chemical active path which is between the grain boundaries. As the boundaries are attacked the stresses are necessarily concentrated, which favors still more intercrystallin penetration. Observation of the progression of cracks in embrittled steel shows that the cracks do not always start at grain boundaries. Often a crack starts by penetration through the outer grain, but once it reaches a grain boundary it progresses along the boundaries.

If another metal like nickel, which in small amounts is completely soluble in the crystal of ferrite, is added to steel the yield point is raised, since the solid solution of nickel in ferrite is stronger than ferrite and slip starts at a higher value which in turn retards the reorganization within the metal. If on the other hand sulfur and phosphorus are increased we have an increase in the strength of ferrite accompanied by a higher percentage of these elements in the boundaries. The yield point is raised but chemical action is not retarded until the yield point is reached, due to the fact that the grain boundaries are much more active.

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[1] Jeffries, "The Science of Metals," p. 78.

In a metal which has been heavily cold-worked, the grains have been completely changed, slips have occurred along various planes, the grains are elongated, the original crystal boundaries have been almost entirely replaced and the maximum strain is on the slip planes of the crystals. A higher stress would appear probable here before the chemical action would progress. It would appear probable that a small amount of cold work would have very little effect on the subsequent embrittling of the metal.

In the pearlitic areas of steel we would expect to find obstacles to the slip of the crystals and as the carbon increases the yield point should increase. The paths of embrittlement cracks should be such as to avoid passing across the lines of pearlite and this is found to be almost invariably so.

CAUSE OF EMBRITTLEMENT IN STEAM BOILERS

It has already been shown that the production of intercrystallin cracks in mild steel can be brought about only by the combined action of stress and proper chemical attack. The limits set for each apply only to the conditions of the laboratory tests and the time of the tests. There is a possibility of a slower action taking place over a period longer than has been tested for, which may lower the stress or the concentration of solution necessary to some degree. Another point to be considered is that the embrittlement tests were run with the metal surrounded by a solution, while in boilers the solution is penetrating between the plates in a thin film and the limits of concentration of solution necessary to produce embrittlement may be altogether different. It is possible to conceive of the stress passing the yield point and the solution reaching the high concentrations used in the tests when one considers that embrittlement only occurs in the riveted areas which are under tension.

Probably in all boiler plates the stresses at the edge of rivet holes occasionally reach the yield point of the metal. It should be remembered that the mathematical theory of elasticity indicates that the localized stresses at the edge of a hole in a plate may be nearly three times the average stress in the plate. This stress concentration at the edge of holes, such as rivet holes, would in itself be sufficient to account for stresses almost high enough to start embrittlement should a solution of proper concentration penetrate to the rivet holes. In addition it should be noted that during the fabrication of the boiler the seams are put together by riveting. The riveting is done hot and, granting only sufficient riveting pressure is used to maintain tight seams, very high localized stress occurs under the rivet heads and around the rivet holes. When one observes the appearance of the plates and straps after they have been removed from a boiler which has been in service and sees the rings around the holes where the straps and plates have been held together by the rivets, there remains little doubt that the metal has been stressed beyond the yield point. When this stress is combined with the stress incident to operation of the boiler, additional local stresses develop which may still more favor embrittlement. The sharp angle which the rivet head makes with the shank combined with the stress forcing the rivet heads apart undoubtedly produces sufficient stress at this point to allow embrittlement to start. It is not uncommon to have rivet heads drop off embrittled boilers long before the plates are embrittled sufficiently to detect cracks. The stresses in plates caused by riveting were studied by R. Bauman,[1] who showed that the plates are stressed locally to values exceeding the yield point even when low riveting pressures were used. H. F. Moore in his address before the American Society for Steel Treating in Spetember, 1925, stated that there was no doubt of the existence of localized stresses which exceeded the yield point of the metal around the rivet holes in riveted sections.

This concentration of stresses exists in all boilers to a greater or smaller degree, yet in the absence of the proper chemical in the boiler water no embrittlement results. If the proper chemical, sodium hydroxide in the absence of much sulfates or carbonates, exists in the boiler water there is a possibility that sooner or later that boiler will become embrittled. The concentration of sodium hydroxide in the boiler seldom surpassed 17,000 parts per million or about 1000 grains per gallon. Laboratory tests show that solutions of this concentration are not detrimental to the steel. There is a possibility that the actual concentration of caustic soda in the vicinity of the seam does sometimes reach a value much above this but still far below 400,000 parts per million or about 23,000 grains per gallon. It is known that sodium hydroxide even of such a low concentration as met with in boiler operation cannot readily be kept under pressure in containers with screw connections. Solutions of other salts can be retained, but with caustic soda leaks tend to develop. The presence of caustic soda under pressure in the boiler tends to produce a seepage of the solution into the seam between the metal surfaces. As the solution penetrates between the plates there is a tendency for concentration due to the steam being released back to the boiler or escaping to the outside. As the concentration progresses, the penetration of the solution further into the seam continues. With time, the concentration becomes sufficient to start embrittlement and if localized stresses are sufficient at the point of concentration embrittlement starts. Often this concentration is reached and embrittlement does not progress, apparently due to lack of sufficient strains and the penetration progresses until a free leak develops. On examination, no presence of embrittlement can be found. At other times embrittlement develops, the plates are weakened and a leak develops which indicates trouble. When the plates are removed from the seams of embrittled boilers soluble salts are found between the plates. This alone would indicate that there must have been considerable concentration in the seams. Another evidence of chemical attack in the seams is the presence of finely divided black magnetic oxide deposited on the surface of the plates.

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[1] Forschungsarbeiten auf Den Gebiete Des Ingenieurewesens. Heft 252, 1922.

The fact that the two main factors for embrittlement must occur simultaneously makes it possible for some boilers to operate under conditions which might point toward embrittlement without developing any distress. The fact that no distress (apparent by general inspection) has occurred is no sign that the boiler is free from danger. An example of this is shown in the case of one installation operating on alkaline water. This plant has been in operation for over eight years and was cited as an example of a boiler operating on alkaline water free from embrittlement. Six months later this plant discovered its distress, which turned out to be a very bad case of embrittlement. The engineer in charge has often since expressed wonder at the fact that one boiler in particular had not exploded, it was so badly cracked.

The real cause of embrittlement in steam boilers can be summarized, therefore, as concentration of caustic soda in the seams in the absence of much sulfate or carbonate together with the existence of localized stresses in the seams.

PREVENTION OF EMBRITTLEMENT

The results of the experimental work on the prevention or inhibition of embrittlement indicates that it can be stopped, but at present these experiments are not complete enough to cite final ratios and concentrations of the chemicals involved. The data collected from plant operation point out that the maintaining of certain ratios of the chemicals will prevent this trouble.

The removal of stresses is not to be considered in a commercial installation, and if it were possible it would not be applied to the many plants already in operation. The practice of inside caulking in drums will, if it can entirely prevent seepage into the seams, tend to retard the occurrence of this type of distress.

The removal of the source of the sodium hydroxide by changing the supply water would be the most effective of any method, but in some instances this is not possible and in others would lead to the use of waters which produce other boiler complications.

The removal of a large percentage of the sodium carbonate with the building up of a sodium-sulfate sodium-carbonate ratio either by the use of sulfuric acid, aluminum or magnesium sulfate all have their disadvantages. The acid treatment is not to be recommended unless installed under the advice and supervision of an expert in such matters and operated under supervision of a chemist or an engineer who understands all the possibilities of its danger. The aluminum and magnesium-sulfate treatment is very effective when used in connection with settling tanks and filters which remove the possibility of scale-forming ingredients in the boiler. These two chemicals are as dangerous, if used in excess, as is the use of acid and should be installed only after a thorough checking of the necessary amounts by a chemist who understands the reactions involved.

TABLE XI.—ANALYSIS, GRAINS PER U. S. GALLON OF WATER TAKEN FROM VARIOUS BOILERS.

	Embrittled			Not Embrittled			
	Denver, Colo.	Champaign, Ill.	University of Illinois, Untreated	Bloomington, Ill. 1925	Chicago, Ill., Treated	University of Illinois, Treated	Illinois Central Railroad, Champaign, Ill.
Sodium Hydroxide, NaOH	176	31.8	60	2	28.2	22	33
Sodium Carbonate, Na_2CO_3	22	10.0	32	10	5.33	9	57
Total Alkalinity as Sodium Carbonate, Na_2CO_3	256	52.0	112	13	42.7	38	174.0
Sodium Sulfate, Na_2SO_4	70	17.8	0.0	200	89.8	90	11
Ratio, Sodium Carbonate to Sodium Hydroxide	0.12	0.31	0.52	5.0	0.19	0.4	1.7
Ratio, Sodium Sulfate to Sodium Hydroxide	0.4	0.55	0.0	100.0	3.2	4.0	0.33
Ratio, Sodium Sulfate plus Sodium Carbonate to Sodium Hydroxide	0.52	0.87	0.52	105.0	3.4	4.5	2.06
Ratio, Sodium Sulfate to Total Alkalinity as Sodium Carbonate	0.27	0.34	0.0	15.4	2.1	2.4	0.63

One point of particular interest is the fact that undecomposed sodium carbonate acts as an inhibitant. Thus if a plant should operate with a high percentage of make-up high in carbonates, the amount of hydroxide formed may be lower since the introduction of fresh carbonates would tend to keep the carbonate-hydroxide ratio high and may in turn inhibit embrittlement. Cases have been cited where the sodium-sulfate to sodium-carbonate ratio has been exceedingly low without any indications of embrittlement and the neutral effect of sodium carbonate is the obvious explanation.

This raises the question as to what ratio should be considered. In the past in the few plants operating on a ratio basis on alkaline waters, it has been customary to consider the ratio of sodium sulfate to the total alkalinity (in boiler waters) expressed as sodium carbonate. This ratio is exceedingly safe since it considers all sodium carbonate as potential hydroxide, but it is misleading in interpreting the operations of certain boilers. Table XI shows the results of analysis of water from the boilers of seven different installations. The first three are from embrittled boilers while the rest represent boilers which are free from trouble. The Bloomington plant is now operating on a sulfate water treated with lime and soda ash and illustrates the high sodium sulfate due to the reaction of calcium sulfate and sodium carbonate during the softening. The Chicago Station and the University of Illinois are carbonate waters treated with sulfuric acid. The ratio of sodium sulfate to sodium carbonate is about 2 in both cases. The Chicago Station working with a lower make-up and a higher steam pressure has a lower amount of remaining sodium carbonate than the University of Illinois water. Assuming that sodium carbonate is also an inhibitant the ratio from actual boiler analysis should be sodium sulfate plus sodium carbonate to sodium hydroxide. When this is the condition, the lower pressure plant with higher make-up is apparently more protected and could in turn work with a lower sodium-sulfate to sodium-carbonate ratio. The ratio of sodium sulfate to sodium carbonate will be misleading in a few isolated cases. The Illinois Central Railroad locomotives operating locally in Champaign on alkaline water illustrates this since the sulfate-carbonate ratio is shown to be as low as 0.63 but the sodium-sulfate plus sodium-carbonate to sodium-hydroxide ratio is over 2 which is much safer and apparently sufficient to stop embrittlement. These illustrations will serve to show that each plant must be considered by itself in order to set any prescription for feed-water treatment in order to inhibit embrittlement.

The information thus assembled, which in some of its phases should without question be extended, is sufficiently conclusive on the fundamental factors to justify its publication at the present stage.

DISCUSSION

Mr. Kerr. MR. H. J. KERR[1] (*presented in written form*).—Knowing something of the time and effort which has been expended by various investigators on this problem, and the conclusions reached by some of them, I feel that the authors are to be sincerely congratulated on the results they have obtained. The public utilities are also to be congratulated on having initiated and sponsored this independent investigation.

In so far as steam boilers are concerned, this type of failure has been constantly before us for some fifteen years. During this period we have seen many kinds of reports explaining away any action of sodium hydrate. These reports have ranged from complaints on the quality of the steel, accuracy of the work, method of manufacture, and have included the term "caustic myth." Fundamentally these explanations have been entirely unsatisfactory, as the same quality of steel, the same type of work and the same method of manufacture have given the best of service, more assured service, where caustic has not been involved, than is usual with power house machinery. Further, the most perfect steel, the most approved construction and the most accurate work have failed with caustic.

In Germany this question has had very considerable discussion since 1912. Two factors led the Germans astray. The first was an extremely poor kind of steel, a kind of steel practically unknown in America, certainly unknown in boiler work. The second was an exceptionally fine piece of experimental work on the type of steel used, but not applicable to the type of steel used in America. German publications to-day indicate, however, that they are also reaching the conclusion that caustic is something more than a myth and it occurs with constructions having all the improvements which their early experiments indicated would eliminate the trouble.

I should like to comment on two points in the authors' paper. In general the stress required to produce the cracks in the caustic solution used has been practically the yield point; ignoring the question of time element in the experiments and in a boiler in service which the authors mention. The specimens tested have been straight bars, so that the stress is actually the same as the calculated stress. In the case of a boiler, where one is dealing with plate pierced by holes, it is well known that the actual stresses at the hole are from

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[1] Babcock and Wilcox Co., New York City.

Mr. Kerr.

2½ to 3 times the average stress as calculated. Therefore, in a joint where the calculated stress is 11,000 lb. per sq. in., there is at the holes a stress of perhaps 33,000 lb. per sq. in., approximately the elastic limit with the type of steel used in boiler work. Just bear in mind that the theoretical allowable stress and, therefore, the stress at the holes, has come about from years of experience which showed that a factor of safety of 5 on the ultimate tensile strength as calculated gave a design which was amply safe. As a matter of fact, in marine service a factor of 4 has given quite satisfactory results, and 3½ is not unknown.

The second point in the authors' paper is the result obtained on the cold-worked specimen. Much has been said about cold work in trying to explain away this "myth." The results shown by the authors on cold-worked samples are therefore worthy of special attention. They show that the stress necessary to produce the trouble in the time available is higher than with the normal plate. It will be appreciated that in boiler design the stress allowed is based on the physical characteristics of the material before it is cold worked. It would therefore appear that cold work, if it affects the problem at all, improves conditions by raising the stress value required.

Until the last few years the problem has not been of serious moment, if the two districts where this type of water occurs naturally (Texas and Illinois-Wisconsin Districts) are excepted. More lately, however, this problem is becoming more generally important, two conditions being responsible. The first is the condenser plant maintaining a certain alkalinity in the boilers in order to be prepared for condenser leakage. In this case the water in the boilers is practically pure condensate with the exception of the sodium carbonate used to maintain alkalinity. The second condition may occur with the use of a zeolite system for treating carbonate waters if no pre-treatment with lime or after treatment with sulfuric acid is employed.

During the years that this trouble has been going on, the boiler maker has not assumed there was nothing for him to do, but on the contrary he has been bringing his workmanship up to standards which five years ago would have been considered impossible, at least commercially. In the case of the manufacturing methods of the company with which I am connected, boiler drum manufacturing methods have been so improved that for some two years drums have been inside calked without outside calking. We believe this construction will prevent the concentration of caustic in the seams and without such concentration apparently the attack cannot take place.

Mr. Applebaum.

MR. S. B. APPLEBAUM[1] (*presented in written form*).—This paper will certainly be of great value, since it creates a better understanding of the cause of boiler failures and of the importance of good boiler operation. The results of former experiments[2] all indicated that the stress is a primary and necessary factor in the production of intercrystallin cracks, but none of these gave any indication as to the magnitude of the stress necessary to produce such cracks. Every discussion of means to avoid cracks of that kind, therefore, resulted more or less in a debate as to the probable magnitude of the stresses required to produce them.

Apparently, there is still some variance of opinion as to the part which caustic soda plays in the phenomenon. Rosenhain and Archbutt presented a paper to the Royal Society, London, in 1919, under the heading "Intercrystallin Fracture of Metals Under Prolonged Application of Stress" in which they advanced an explanation for this not very common failure. Later, in April, 1921, at a joint meeting of the Faraday Society with other English societies, Rosenhain in an introductory address to the general discussion of "The Failure of Metal Under Internal and Prolonged Stress," expressed as his opinion that if a chemical action upon the intercrystallin cement could occur, then the products of the chemical action would soon plug the narrow fissures between the crystals and stop the action, unless there was viscous or visco-elastic movement, due to stress. The authors' experiments now confirm this view experimentally, because they clearly show that the steel in contact with chemical solutions does not crack so long as the products of the chemical action are not displaced or broken as a consequence of stress and that such displacement or breaking of the products of chemical action takes place only if the stresses exceed the yield point.

Rosenhain's conclusions as to the production of cracks, based upon experimental work, were that the chemical action is essentially an accelerating one, but that the cracking takes place even under conditions in which the possibility of chemical action is excluded. This view is confirmed by cases of intercrystallin fractures found in steel not exposed to caustic soda at all, such as in bridges and in other steel structures,[3] in shafting,[4] in boiler plates outside of the seams,[5] in steel fire tubes in smoke boxes of locomotives,[6] in boilers using feed

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[1] Permutit Co., New York City.

[2] See for example, Jones, Faraday Society, Vol. XVIII; also Bureau of Standards; also *Chemical and Metallurgical Engineering*, February 11, 1924.

[3] Rosenhain, Faraday Society, 1921, p. 3.

[4] Baumann, B.R.V., 1925, No. 15.

[5] Baumann, *Ibid.*; also J. E. Fletcher, Faraday Society, 1921, p. 159.

[6] Fowler, Faraday Society, 1921, p. 82.

Mr. Applebaum.

water containing no caustic soda,[1] in steam spaces above the water line of boilers,[2] and in many cases of non-ferrous metals.[3] It has also been reported by Steam Boiler Users Associations abroad that the cracking has been found in riveted seams of steam boilers in which the recommended sulfate-carbonate ratio was present.

To the above, there must furthermore be added all cases of embrittlement produced by stressing steel at blue heat if the term embrittlement is understood to refer to fractures without plastic deformation produced by a stress below the ultimate tensile strength of the original material. For example, Ridsdale as early as 1898[4] refers to intergranular weakness which can be produced by hammering steel at blue heat. More recently Baumann did further work along similar lines.[5] Many brittle rivets may have been produced in this manner. Spencer[6] refers to other causes which may embrittle rivets. All of these cases clearly show that brittleness may be produced by stress alone in the absence of any chemical action.

Mr. Parr's experiments should also prove of benefit in clarifying the issue as to the strength of caustic soda solution (20,000 grains per gallon) necessary for the accelerating effect. This strength obviously does not exist in the main body of the boiler water and therefore the question of the possible concentration to such strengths inside boiler seams becomes a question of prime importance. Both practical experience as well as experimental work done so far tend to disprove any appreciable differences in concentration. It is well known that boilers, previously made tight by precipitating scale into the leaking parts, will begin to leak again if subsequently operated with a different water containing the scale-forming constituents in amounts below their saturation point. This is due to the fact that the old scale, which filled the interstices, is gradually leached out by the water not saturated with these substances. This proves that any space under the water line which is subject to concentration is also subject to diffusion or dilution. Experiments in the laboratory with caustic-soda solutions confirm this. In the authors' tests[7] mention is made of a special steel jacket placed closely about the specimen in the container in order to precipitate sodium sulfate around the specimen. The authors stated that: "In this manner conditions approaching

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[1] Powell's discussion of A. E. White's paper, *Transactions*, Am. Soc. Mechanical Engrs., December, 1920.

[2] Stromeyer, Iron and Steel Inst., 1907; also Quack, Verein Deutscher Ingenieure, 1925, p. 1091.

[3] Rosenhain, Faraday Society, 1921.

[4] Iron and Steel Inst., Vol. I, pp. 220 *et seq.*

[5] Verein Deutscher Ingenieure, Res. 252.

[6] *Boiler Maker*, February, 1926.

[7] See p. 71, third paragraph.

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those of concentration in the seam of a boiler were obtained." The conditions, however, are quite different, because the concentrations inside and outside the steel jacket were apparently the same in the case of these tests, whereas in a boiler the concentration of the sodium sulfate in the main body of the boiler never should reach the saturation point.

Another factor which deserves consideration is the calcium and magnesium content of the natural well waters in the geographical districts mentioned. For example, the analysis of Champaign water (Table II of the paper) shows that out of a total solids content of 23 grains per gallon, about 16 grains consisted of calcium and magnesium salts. These salts are very much less soluble than caustic soda and before a concentration of 20,000 grains per gallon can be reached in the seams more than 20,000 grains per gallon of calcium and magnesium salts must have precipitated. That calcium and magnesium salts do in fact precipitate in seams was proved by analyzing samples of deposit taken out of a seam of an embrittled boiler using this water. Why did these calcium and magnesium precipitates not inhibit the action, if a protective mechanical coating is all that is required?

In the second paragraph on page 70 of the paper, it is stated that the specimens embrittled in the laboratory show a large number of intercrystallin cracks progressing into the strained portion. It is not stated, however, whether the final fracture itself was intercrystallin. Mr. Jones,[1] who, as stated above, made experiments similar to those described in the present paper, stated the following:

> "In the case of the more concentrated solutions some intercrystallin cracks were first formed, but ultimate failure generally occurred by fracture through the crystals."

It would be of interest to know whether the authors can confirm this statement from their experiments.

Good engineering practice cannot regard boiler parts subjected to stresses beyond the yield point with equanimity. The view that such excessive stresses must be accepted, because said to be unavoidable, must be questioned in view of the many investigations which show the danger connected therewith. Such matters as fatigue,[2] the behavior of metals under prolonged stress at elevated temperature,[3] the influence of deformation of steel at different temperatures upon the impact resistance,[4] the influence of shocks and intercrystallin

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[1] Faraday Society, December, 1921.

[2] University of Illinois, *Bulletins Nos. 124, 136, 142* and *152.*

[3] H. J. French, "Methods of Test in Relation to Flow in Steels at Various Temperatures," see p. 7.

[4] Richardson and MacNutt, Am. Soc. Steel Treating, April, 1924; also Körber and Pomp, K.W.I., 925.

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fatigue,[1] and the determination of the elongation of thick boiler plates[2] must be considered in conjunction with the authors' results in determining the causes of failures. The statistical evidence which can be expected to accumulate in the future from the recommendation in the A.S.M.E. Boiler Code, Section "Care of Boilers," namely, to investigate all seams which leak and do not remain tight after proper calking, should also increase our knowledge as to the various causes of the failures under discussion.

Steps have already been taken to abandon riveted seams for high-pressure boilers. This construction has also been adopted abroad, in part at least, for boilers of medium pressures. Its adoption makes it possible (1) to avoid the necessity of accumulating large steel masses in seams which always give rise to non-uniformity of heat conduction, and (2) it will make possible the proper annealing of finished drums after their fabrication, which is not possible with riveted drums, so that the undesirable internal stresses due to manufacture can be eliminated or at least reduced to a permissible limit.

It may be of interest to mention that abroad not only forged but also hammer-welded drums have come into use, which are subjected to a high hydraulic pressure after proper annealing, in order to secure a truly circular shape and then are again annealed, in order to remove the stresses introduced by the hydraulic pressure. The ends of these drums are then turned in a lathe, as are the flanges of the dished heads after annealing in order to secure a proper fitting. Riveted circumferential seams produced in this manner are the only seams in the drum.

Mr. Grunert.

MR. A. E. GRUNERT[3] (*presented in written form*).—In three of the large generating stations of the Commonwealth Edison Co. in Chicago the feed water make-up is treated with a Zeolite process. The source of supply at Crawford Ave. is the Drainage Canal, at Calumet the Calumet river and at Northwest the north branch of the Chicago river. The soluble constituents of these waters are practically the same in quantity and character. There are submitted in Table I typical analyses of the waters at Crawford Ave. at each stage of operation. The untreated water is what would ordinarily be classed as a calcium and magnesium carbonate water and relatively low in sulfate. It is Lake Michigan water which has been altered by river conditions in Chicago.

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[1] T. M. Jasper, "Report on Cause of Failure in Metal of A. O. Smith Cylinder Tested in Repeated Stress Under Repeated Hydrostatic Loading," *Journal*, Am. Welding Soc., January, 1926.

[2] Baumann, B.R.V., 1925, Nos. 6, 7 and 8.

[3] Commonwealth Edison Co., Chicago, Ill.

Mr. Grunert. In the older plants of this company this water has been used as boiler feed make-up for years; on certain boilers since 1903. Under these conditions there has been no trouble with boiler plates but the boilers scale badly and frequent turbining is necessary.

TABLE I.—TYPICAL FEED WATER CONDITIONS AT CRAWFORD AVE. STATION, COMMONWEALTH EDISON CO., CHICAGO, ILL.

(Units in grains per gallon.)

	Raw River			Filtered River			After Permutit			After Acid			Feed Water[b]	
	1	2	3	1	2	3	1	2	3	1	2	3	1	2
Suspended matter[a]	2.79	3.03	1.60	trace	trace	trace	trace	trace	trace	trace	trace	trace	trace	trace
SiO_2	1.12	0.40	0.82	0.38	0.46	0.30	0.16	0.39	0.59	0.46	0.73	0.41	0.03	0.10
$FeO_3 \div Al_2O_3$	0.07	0.23	0.05	0.06	0.03	0.06	0.11	0.15	0.05	0.11	0.19	0.10	0.01	0.02
$NaCl$	2.91	0.50	1.55	1.94	1.44	1.76	2.04	2.65	1.76	1.94	2.65	1.55	0.19	....
Na_2SO_4	0.81	....	0.13	2.67	....	0.04	2.63	2.72	2.92	11.29	6.13	10.27	0.23	0.19
$MgCl_2$	....	0.71	....	....	1.06	....	....	....	....	....	....	....	....	....
$NaHCO_2$	....	....	....	0.61	....	....	5.58	6.05	5.97	0.68	2.45	0.99	trace	....
$CaSO_4$	....	0.12	....	....	1.91	0.66	....	....	....	....	....	....	0.08	0.02
$CaCO_3$	5.99	4.60	5.02	5.52	3.59	4.40	0.06	0.06	0.04	0.10	0.20	0.06	....	0.01
As $Ca(HCO_3)_2$	9.71	7.46	8.14	8.94	5.82	7.13	0.10	0.16	....	0.16	0.32	0.10	....	0.02
$MgSO_4$	2.26	2.45	2.02	....	0.65	2.45	....	....	....	....	....	....	0.23	0.09
$MgCO_3$	1.01	....	0.87	2.24	....	....	0.12	0.26	0.08	0.34	0.10	0.14	....	....
As $Mg(HCO_3)_2$	1.75	....	1.51	3.89	....	....	0.21	0.45	0.14	0.59	0.17	0.24	....	....
Organic matter, etc	1.92	3.59	1.72	1.88	3.76	1.86	1.95	2.21	1.44	1.80	3.35	1.34	0.37	0.24
Total solids	18.88	15.63	13.78	15.30	12.90	11.53	12.65	14.49	12.86	16.72	15.80	14.86	1.14	0.67
Dissolved	16.09	12.60	12.18	15.30	12.90	11.53	12.65	14.49	12.86	16.72	15.80	14.86	1.14	0.67

[a] Mostly clay.

[b] Feed water would contain normally about 4 per cent of acid neutralized Permutit water and about 0.50 per cent raw river condenser leakage.

TABLE II.—TYPICAL BOILER WATER CONDITIONS AT CRAWFORD AVE. STATION, COMMONWEALTH EDISON CO., CHICAGO, ILL.

(Unit in grains per gallon.)

	Steaming Hours	Total Solids	Sodium Sulfate	Alkalinity		Sodium Sulfate-Sodium Hydrate Ratio	Sodium Sulfate-Total Alkalinity as NaOH Ratio
				Sodium Hydrate, $NaOH$	Sodium Carbonate, Na_2CO_3		
No. 1	672	188.60	86.97	25.10	8.31	3.46	2.77
No. 2	714	205.54	99.20	33.31	4.43	2.98	2.70
No. 3	413	223.30	93.27	27.86	11.84	3.35	2.54
No. 4	512	136.73	63.25	20.53	8.76	3.08	2.33
No. 5	657	157.44	76.36	20.02	8.33	3.81	2.90
No. 6	566	255.98	122.62	36.81	9.57	3.33	2.78
No. 7	1296	209.47	115.09	26.41	6.32	4.36	3.69
No. 8	280	250.73	93.69	25.57	8.33	3.66	2.94
No. 9	390	196.15	98.37	26.55	7.64	3.71	3.04
No. 10	406	108.57	58.23	11.71	7.01	4.97	3.42
No. 11	290	137.44	66.09	17.17	6.72	3.85	2.98
No. 12	411	171.43	92.10	23.30	6.55	3.95	3.26
No. 13	562	155.66	76.95	16.88	6.34	4.56	3.55
No. 14	820	264.86	150.38	30.71	9.43	4.90	3.98
No. 15	726	165.34	82.56	25.45	7.58	3.24	2.65
No. 16	1109	170.99	81.97	23.80	11.97	3.44	2.50

The water conditions at the other plants mentioned, where make-up is treated, are somewhat altered as inherent to the Zeolite process of softening water. This treatment gives a water high in

Mr. Grunert.

sodium carbonate which becomes largely converted to caustic soda in the boiler. This condition of high causticity and relatively low sulfate is not considered desirable. This of course is the principal subject matter of the recent experiments by Parr and Straub. It has apparently become somewhat controversial and until this has reached a stage of conclusive verification our attitude is one of caution and insurance. It is felt, however, that the results of the Parr and Straub experiments are such that they cannot be neglected as a possible safety factor in the operation of large boiler units under relatively high pressures with water conditions as stated.

The precautions taken in our plants where make-up is so treated is partial neutralization by sulfuric acid regulated in such a manner that the sulfate-caustic ratio is maintained in excess of 3 to 1 as recommended. The water conditions after acid neutralization and in the boiler are submitted in Tables I and II.

In addition the manufacturer of the boilers at Crawford Ave. has given the matter of construction considerable thought from the viewpoint of embrittlement. As a result all seams are calked on the inside, without any calking on the outside, the idea being to minimize local caustic concentrations.

The operation in connection with partial acid neutralization of the Zeolite water is simple and relatively inexpensive. It furthermore does not interfere with the advantages gained by this form of water treatment under our particular conditions. The boilers are kept clean, which is the principal result desired. The blow-down of the boilers is controlled by the standard alkalinity test, the maximum allowed being an amount arbitrarily taken as suitable for our conditions.

The boilers longest in service under these conditions are the first six boilers at Calumet Station. They have been in continuous operation with Zeolite make-up water partially neutralized for over 3½ years. The sulfate-caustic ratio is being maintained in excess of 3 to 1. During all that period there has never been a tube removal due to scale conditions, the tubes have never been turbined and there is to date no indication of embrittlement in any form. Whether some form of embrittlement would have occurred if the make-up had not been acid treated we are in no position to state definitely. It is our policy to take all the reasonable precautions recommended in so far as they are practical. We at least have some ease of mind on this question and feel that the safety and insurance feature of this practice is good business.

Mr. Savidge. MR. R. W. SAVIDGE.[1]—I should like to ask Mr. Parr if he has any theory in regard to the chemical reaction between the iron and caustic soda?

Mr. McAdam. MR. D. J. MCADAM, JR.[2]—I am especially interested in this paper, because it touches the edge of the field of investigation described in my paper covering corrosion-fatigue.[3] The authors have discussed the effect of combined chemical action and corrosion. In my own paper I discuss the effect of combined chemical action and a range of stress. I think that the authors possibly have over-emphasized the distinction between corrosion cracks and embrittlement cracks. It seems very probable that no sharp distinction should be made. As the authors show, embrittlement cracks are due to continued chemical action and stress, whether that stress be corrosion or chemical action by sodium hydroxide. In our work on the effect of combined corrosion and fatigue, we have made a few experiments on ingot iron, and we find that the cracks are at least partly intercrystallin; also the cracks due to combined corrosion and internal stress of non-ferrous metals are usually intercrystallin.

I think that the authors' paper touches one phase of a very broad subject which we are now entering, and one which we should have entered as soon as we encountered such phenomena as season cracking. I should like to suggest that the authors consider the possibility that there is a range of stress in boilers in service which, when combined with chemical action, may be responsible for failures.

Mr. Patterson. MR. C. T. PATTERSON.[4]—The authors are to be congratulated on their contribution to the study of the effect of strong caustic soda on steel under tension and heated; also in the development of a treatment which has prevented embrittlement in one type of boiler feed water. But let us not overlook parallel phenomena which may have occurred. Artesian water frequently contains more dissolved gases than surface water. The neutralization of a large amount of bicarbonate of soda by pouring sulfuric acid into the water will release a large amount of CO_2 which will tend to sweep out dissolved gases and thus make the water less corrosive. Let us also note that corrosion is always evident in boiler plate embrittlement crackings, that iron oxide is found in the cracks, and that caustic soda has long been known and used to prevent corrosion, and when it is not sufficient or when oxygen is too plentiful, this should be recognized. Further, since

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[1] Head Chemist, Union Pacific Railroad Co., Omaha, Nebr.

[2] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[3] D. J. McAdam, Jr., "Stress-Strain-Cycle Relationship and Corrosion-Fatigue of Metals," see p. 224.

[4] The Solvay Process Co., Syracuse, N. Y.

Mr. Patterson. we know the great affinity of strong caustic soda solutions for fresh water, let us be sure that concentrations of even 350 g. per liter or more could form in crevices in a boiler while generating steam under 150 lb. or more pressure and with a turbulent circulation of fresh water past the inlet to the crevice. Finally, in the tests reported in the present paper, let us consider the great difference in temperature of testing the steel in distilled water generating steam at 60 lb. pressure and 400 g. per liter of caustic solution generating 60 lb. pressure, and also the effect of air and water on steel under severe tension at these temperatures, and the possible reinforcement of tightly crystallized salt inside the steel jacket only $\frac{1}{32}$ in. larger than the test piece.

Mr. Speller. MR. F. N. SPELLER.[1]—This very interesting paper suggests many questions but I will mention only one or two points which seem important and are apparently not covered. In the first place hydrogen has been reported as being generally found in steel which shows caustic embrittlement. That being the case, it seems that this type of embrittlement may be connected with that class of chemical corrosion that is accompanied by occlusion of hydrogen in the metal, as in acid attack. The action is probably accelerated by the presence of incipient cracks, strain, and possibly by the electrolytic action of local concentration cells. I should like to ask Mr. Parr if the possibility of the accelerating effect due to differences in concentration of the solution in the cracks and in the boiler has been investigated, and also whether he has found in his laboratory tests that embrittlement occurs after the metal has been previously stressed beyond the elastic limit and the stress released.

Mr. Risteen. MR. A. D. RISTEEN.[2]—I should like to ask a question, but I am specially anxious that it should not be interpreted even as a remote criticism of this excellent paper. For a great many years, of course, sodium carbonate (soda ash) has been used in boilers for the treatment of scale conditions, and in a great many cases—not so much as formerly, yet in a great many cases still—it has been unintelligently thrown in until the boiler would perhaps even foam. Now it seems to me that we should have seen some evidence of steel embrittlement in connection with this excessive use of soda ash. Perhaps we have; I am only asking for information. I know, of course, that there were a great many cracks before the day of the butt-strap joint. We had the so-called lap joint crack, for example; but that would seem to be explained sufficiently by other conditions. I should like to know if Mr. Parr, or somebody else, can throw a little light on that point of the excessive use of soda ash in the old days.

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[1] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

[2] Director of Technical Research, Travelers Insurance Co., Hartford, Conn.

Mr. Parr. MR. S. W. PARR.—I do not know whether I can touch on all of these points raised; I should like to make one statement as a blanket response, which is this: do not think for a moment that we know all about embrittlement. We have established what we think are a few facts. Like all other investigations, every new item opens up a great many more questions, and I am very glad to say that the funds which have been put at our disposal have been extended for another year or more, so that I hope we will find out answers to many of these questions that have been asked at this time. If I were to proceed in reverse order, I might say that the excessive use of soda ash would need to be studied with reference to the particular circumstances. For instance, in one of our surveys we found the use of soda ash in rather moderate amounts but in connection with a pure water and with disastrous results; that is to say, a treatment which would have made a bad water good, in the case of pure water the soda ash, inadvertently or ignorantly put in, made that good water very bad and embrittlement certainly was due to the use of a moderate amount of sodium carbonate. I think that the question of the use of sodium carbonate would have to be determined by reference to the specific case in hand. Sodium carbonate is used by many railroads. If the water to start out with is high in sulfate, it would be impossible for the amount of sodium carbonate to dominate the situation to such an extent as to bring about the conditions which at the present time we think are essential for producing embrittlement. In general I think that is as near as I can come to answering that question at the present time.

Now this matter of hydrogen and hydrogen embrittlement—two of the questions have related to that. We have not tried to determine or to analyze the mechanism of the reaction that goes on between the crystallin boundaries and the chemical, assuming that Professor Matthews, of the Massachusetts Institute of Technology, who is working in that specific field, will give us the information. However, in our former work, where we sealed up caustic with samples of steel and kept them at a certain temperature for a certain length of time, we always found a positive amount of hydrogen upon opening the steel containers. There is no question but that hydrogen is a part of the reaction between sodium hydroxide and iron, with hydrogen as a by-product. We have, then, not only an explanation for the iron oxide that forms, but we have the question raised as to what effect this hydrogen, in its nascent stage, may play in further reactions. But we should divorce the idea of hydrogen embrittlement from the phenomenon of caustic embrittlement. There may be, there doubt-

Mr. Parr. less is, a relation between the two, and doubtless hydrogen plays a part in both types of phenomena, but that type of embrittlement due to hydrogen and which is designated as hydrogen embrittlement will disappear when the metal is relieved of the hydrogen by heat or otherwise, and the embrittling effect due to the presence of hydrogen disappears. This is not the effect that we get in the so-called embrittlement produced by caustic.

Mr. Applebaum refers to the matter of the impossibility of a concentration within the seams up to the point where this reaction would be explained. Now that is simply a matter of difference of opinion. I take it that he does not see the thing my way, and I certainly do not see the thing his way. In fact, the study of these encrustations shows them to be explainable along the lines I have given, that is to say an interspace concentration of caustic up to the point where chemical reaction is set up. I cannot quite understand his reference to the composition of the Champaign water as given in Table II of the paper; the calcium carbonate is given as 9.75 grains to the gallon; the magnesium carbonate as 6.24 grains to the gallon. Now those carbonates cannot get into the cracks, they are readily precipitated in the ordinary reactions in the boiler and at once those substances are thrown out as sludge. I cannot see how they can be a constituent part of the compound that forms in the cracks. On the other hand, so far as the chemical analysis of those waters is concerned, I do not see how anything else than salts of soda, either sulfate or hydroxide or chloride, can be present in the seam.

Answering Mr. Patterson's question, there certainly is no turbulence in these cracks whereby the water that gets in is washed out. If it gets in, I am inclined to think that the only thing that can happen to it is to reach a concentration by discharge of steam, the residue of salt remaining in the cracks.

Mr. Kerr raises the question of the time element in those artificially produced cracks as compared to the time element in the case of boilers in actual use. I am glad he brought out this point, because I am quite convinced that the time element in these artificial cracks is quite different from the conditions existing in the boiler, that is to say, things might occur over a long period of time as in the case of a boiler in service which would be somewhat different from these quick, rapid tests that are reproduced artificially. It is a little like the corrosion tests carried out by submerging a metal in acid, while they cannot be interpreted in terms of actual use, they are still of value as giving comparative information. In the same way, these artificial conditions afford an index of what is going on in the case of boilers in actual use.

SOME DEFECTS WHICH HAVE BEEN FOUND IN LARGE CARBON-VANADIUM FORGINGS—THEIR CAUSES AND PREVENTION

By O. B. Schultz[1]

Synopsis

In the past, very little attention has been given by the consumer to the process of melting and refining forging steels. This paper discusses the quality of carbon-vanadium forging steel as influenced by (*a*) the manufacturing process, (*b*) forge shop practice, and (*c*) heat treatment.

The usual method of inspection subsequent to manufacture is useless, and a waste of both time and money.

If the melt of steel has been properly made under great care and proper supervision the problem of heat treatment will be minimized and the product will withstand the severe service demanded of this class of material.

A series of photomicrographs is shown illustrating the various conditions encountered in normalized carbon-vanadium forgings.

This paper is a plea for the recognition of the importance of manufacturing processes in the production of steel for steam locomotive forgings, particularly those made of basic open-hearth carbon-vanadium alloy, the use of this class of steel having steadily increased since 1910.

In an effort to increase their hauling capacities, steam locomotives have reached about the limit in weight on drivers for present track conditions. Therefore, it is necessary to make savings in weight wherever possible by the use of lighter sections of greater tensile strength. Alloy-steels are ideal for this purpose, and since heat-treating problems increase with the complexity of the alloy, the simpler alloy-steels are more adaptable to the facilities usually found in railroad shops.

It is rather deplorable to state that due to the results obtained by the use of carbon-vanadium steel, many turned to its use without any consideration of the quality of the material, so long as it was called "Vanadium-Steel." Manufacturers who had no experience in making alloy steels rushed into its production, and the natural result was that some very bad steel was produced. Forging plants attempted to make carbon-vanadium forgings with the same practice as had been used for the production of medium-carbon steel forgings. The natural result of this mal-practice is, that with some of the railroad companies, this class of steel is not in good repute. Conditions, however, in these respects are continually improving, and to-day there are several manufacturers who are doing everything they can to make the best possible product. Forging methods are steadily being improved, and modern heat treating furnaces are capable of very close control. All of these factors are absolutely essential to the production of sound forgings which will withstand the severe service demanded of them in railroad use. In the majority of cases, carbon-vanadium forgings are used in the normalized condition. The normalizing process consists of heating the forgings to a temperature of 1550 to 1600° F. (845 to 870° C.), maintaining this temperature a sufficient length of time to insure the entire forging having a uniform heat, and then removing the forgings from the furnace to cool in still air, protected from draughts and rain or snow. The results of tension tests and the microstructure of specimens taken from the heads and bodies of the forgings are uniform when the steel has been properly made and the forging properly done.

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[1] Chemist and Engineer of Tests, Lima Locomotive Works, Inc., Lima, Ohio.

The usual specifications for carbon-vanadium billets or blooms cover only the chemical requirements, with subsequent surface inspection to insure obtaining billets or blooms free from surface defects such as seams and slivers, or too deep chipping. The process by which the steel is to be made is sometimes stated, but the actual making of the steel is left to the discretion of the manufacturer, which is as it should be. But, take the case of the manufacturer who is not entirely familiar with alloy-steel practice. In many cases, the purchaser and the producer can seek each others advice with mutual benefit and thus obtain a satisfactory product. It is, therefore, essential that the purchaser familiarize himself with the steel he is attempting to use, in order to protect himself and his product. The usual inspection by the purchaser at the mill is entirely a waste of time and money, for almost all specifications for billets have a clause which provides that billets which show injurious defects while being finished by the purchaser will be rejected. Since the manufacturer must stand the loss of the material and pay the shipping charges both ways, his mill inspection is usually more severe than that of the ordinary inspector.

It has been found that two pieces of steel from different melts and mills, with almost identical analyses, respond entirely differently to forging and normalizing. Having so many concrete examples of such varying results, it was decided to investigate them with the object of determining means for their prevention, since it was found that forgings with extraordinary variances in physical results were the ones which were proving defective in service.

Any steel, be it plain carbon or alloy, is subject to inherent defects, in addition to those defects which can be so easily produced during the fabricating process, and it early became apparent that some of the few defective forgings which were produced and which failed in service, failed on account of defects which could not have been forged into them. At this point the microscope plays a very important rôle, and it is an invaluable aid in determining the causes of defects.

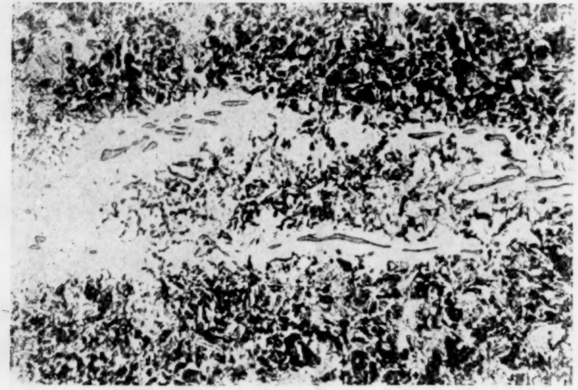
Figure 1 shows the structure of a forging which failed on account of weakness due to non-metallic inclusions. These inclusions are found in the grain boundaries, and their tendency is to weaken the structure by providing a starting point for a progressive fracture. In carbon-vanadium steel the usual manganese range is 0.60 to 0.90 per cent, and it is considered good practice to add an amount of ferro-manganese, equivalent to about 0.25 per cent of the total weight of the charge before going ahead on the heat. This tends to remove the oxides from the bath. If the charge melts high in carbon and it is necessary to add ore to the heat in order to make time on the furnace, care should be exercised that the heat is not ored too close, and that the ore is added as soon as possible after the heat has become sufficiently melted. No ore should be added to a heat later than at least two hours before tapping time. This is to insure as near as possible a bath free from oxides at the time of tapping. Care should be exercised in using fluorspar, since an excessive amount thins the slag, and lessens its affinity for oxides. The percentage of limestone in the heat should never be less than 10 per cent of the charged metal, and better steel is insured if 11 or 12 per cent is used. This makes a very basic slag which in turn prevents rephosphorization and also helps to remove the sulfur. The slag should contain at least 55 per cent of active bases.

The most important factor in obtaining a normal heat of steel is the conditioning of the bath. Time must be given for the necessary reactions to take place in the furnace and excessive "oreing" and "pushing" the furnace does not hasten them. A sharp-working furnace is necessary for the making of good steel, and if the heat is rightly proportioned when the charge is made up, by the time the heat is melted it will contain sufficient carbon to insure a normal drop without the addition of ore. At this time in the process of making the heat the slag should appear white and "creamy." Heats having red-looking slag are undesirable. All of the lime should be up some time before going ahead with the heat. If the lime is not all up it can usually be determined by small blue flames coming out in various places below the level of the bath. Proper working of the heat and conditioning of the bath may be insured by allowing the heat to bring up its own lime. Continual punching at the bottom of the furnace to bring up the lime and hasten the progress of a heat usually results in starting a boil on the bottom, thereby filling the bath more or less with impurities. Great care should be taken to have the heat at the right tapping temperature by the time the heat is properly conditioned. By having the heat of steel very hot at this time, the metal will be quite thin which gives the impurities a chance to rise freely to the slag. If a heat has to be delayed after it is properly conditioned on account of not being at the proper tapping temperature, the steel is very apt to become over-oxidized. It is very difficult to eliminate this condition, and it produces a very inferior grade of steel. There is an old saying among steel melters that a heat is ready to be tapped but once.

Additions to the melted steel in the ladle are often sources of serious segregrations. This is particulary true when the tapping temperature is not sufficiently high. The temperature of a vanadium heat must be not less than 2980° F. (1638° C.) at the time of tapping, or the melted steel will not assimilate the ferro-vanadium. The best results are obtained by having a medium flow of metal from the furnace and the addition of the ferro-vanadium at a uniform speed. All of the ferro-vanadium must be in before the slag comes. If a swirling motion can be obtained in the ladle, segregation of vanadium is less apt to occur. The ferro-vanadium should be uniform in size and not in the form of dust, or losses will occur. It should never be necessary to add more than $\frac{3}{4}$ lb. aluminum to each ton of steel.

Recarburization in excess of 0.03 per cent should not be permitted. During a period of over three years in which heats have been used which have not been recarburized, and many which have been recarburized up to 0.03 per cent, it has been impossible to distinguish any difference in structure and physical results obtained. The limit of recarburization of 0.03 per cent was set to insure the making of the steel in the furnace and not in the ladle.

The most serious defects in carbon-vanadium forgings are checks, and one of the causes of these defects is improper heating in the soaking pit. Too rapid heating of a comparatively cold ingot produces internal bursts, and careless heating produces burnt edges. In the subsequent rolling, these conditions are exaggerated. In order to prevent the formation of such checks great care and skill must be used by



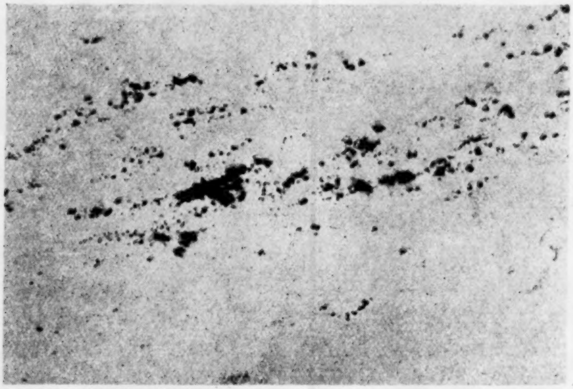
Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	53 000
Tensile strength, lb. per sq. in.	69 350
Elongation in 2 in., per cent	None
Reduction of area, per cent	None

FIG. 1.—Abnormal Steel Due to Poor Conditioning of Bath, Main Rod No. 940, Normalized. Etching Reagent, 2-Per-Cent Nital[1] (× 100).



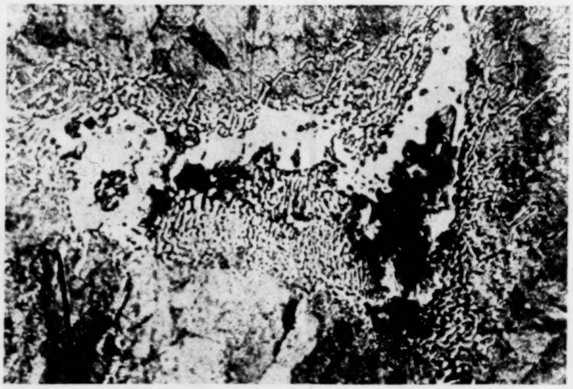
Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	65 000
Tensile strength, lb. per sq. in.	113 000
Elongation in 2 in., per cent	12.0
Reduction of area, per cent	22.3

FIG. 2.—Segregation of Alumina, Probably Caused by Improper Tapping Conditions. Main Rod No. 949, Normalized. Unetched Surface (× 100).



Objective

Bausch and Lomb 4 mm. Achromatic.

Light

Arc.

FIG. 3.—Illustrating Burnt Edges as Heated in the Soaking Pit, Bloom as Rolled. Etching Reagent, 2-Per-Cent Nital (× 500).

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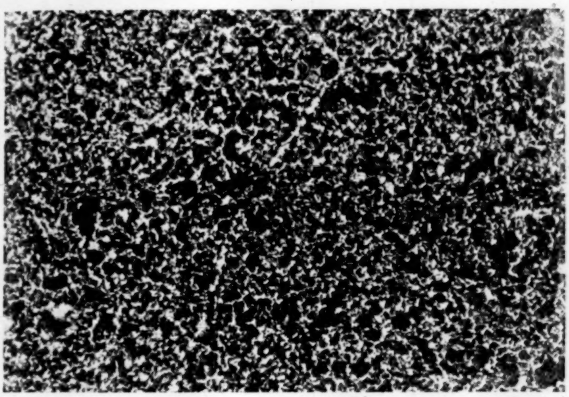
[1] Nitric acid and alcohol.



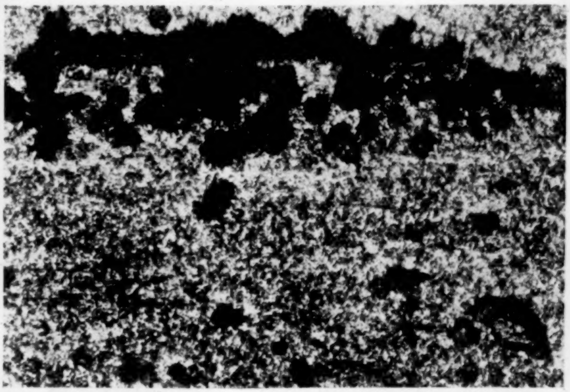
Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	59 850
Tensile strength, lb. per sq. in.	98 050
Elongation in 2 in., per cent	15.5
Reduction of area, per cent	26.1

FIG. 4.—Overheated Structure in Finished Forging, Main Rod No. 845, Normalized. Etching Reagent, 2-Per-Cent Nital (× 100).



Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

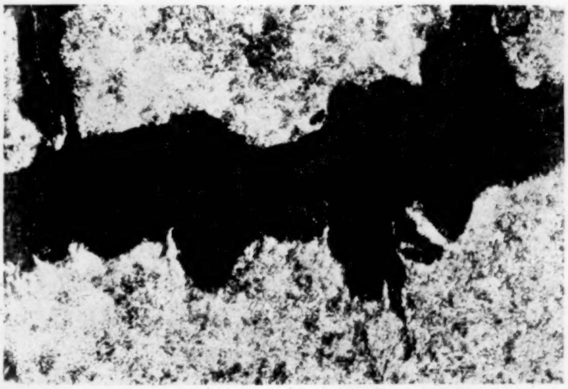
Objective

Bausch and Lomb 4 mm. Achromatic.

Light

Arc.

FIG. 5.—Checks Caused by Pipe, Main Rod No. 950, Normalized. Etching Reagent, 2-Per-Cent Nital (× 500).



Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

Objective

Bausch and Lomb 4 mm. Achromatic.

Light

Arc.

FIG. 6.—Checks Caused by Too Fast Heating, Main Rod No. 961, Normalized. Etching Reagent, 2-Per-Cent Nital (× 500).

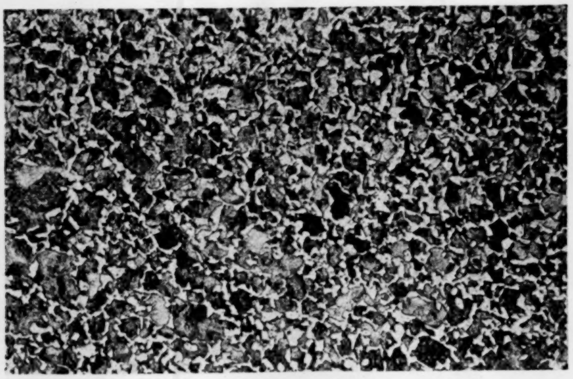


FIG. 7.—Proper Structure as Finished Under the Hammer, Main Rod No. 985. Etching Reagent, 2-Per-Cent Nital (× 100).

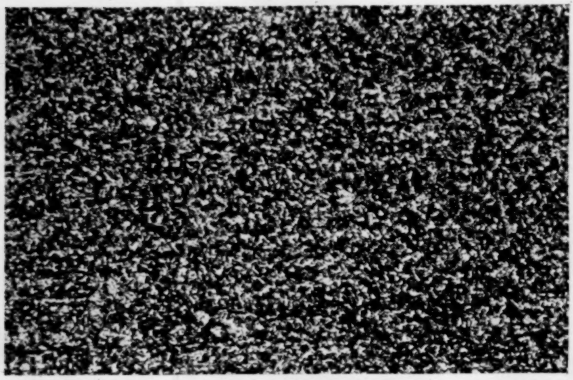


FIG. 8.—Structures of Cold-Worked Forging, Main Rod No. 993. Etching Reagent, 2-Per-Cent Nital (× 100).

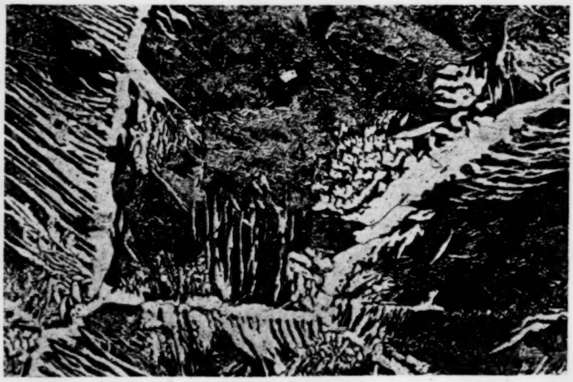
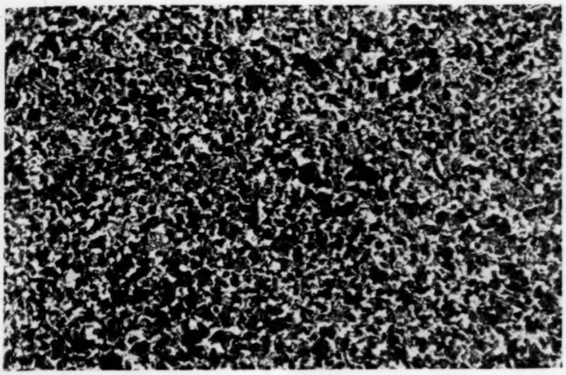


FIG. 9.—Illustrating Overheated Structure as Heated in the Soaking Pit, Bloom as Rolled. Etching Reagent, 2-Per-Cent Nital (× 100).



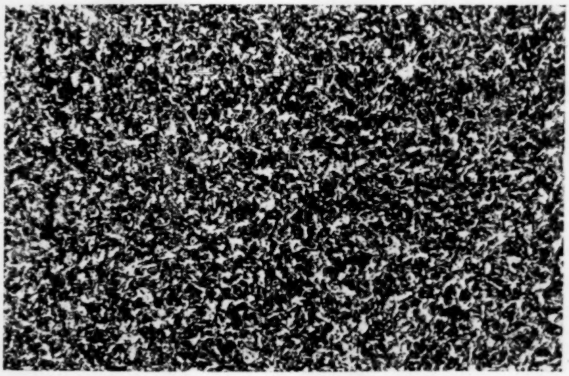
Treatment

Held 8 hrs. at 1600° F. (871° C.), cooled in air.
Held 8 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	67 700
Tensile strength, lb. per sq. in.	102 950
Elongation in 2 in., per cent	28.5
Reducation of area, per cent	52.6

FIG. 10.—Ideal Structure, Normalized Carbon-Vanadium Forging, Main Rod No. 890, Normalized. Etching Reagent, 2-Per-Cent Nital (× 100).



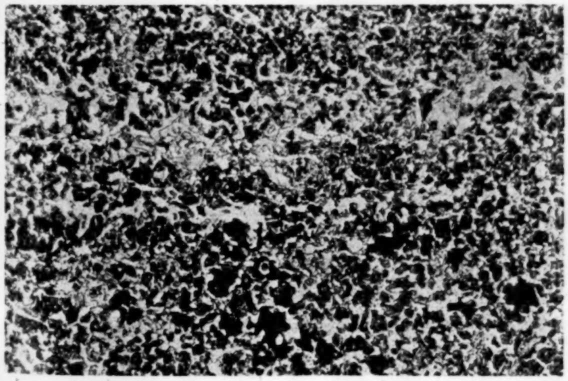
Treatment

Held 5 hrs. at 1600° F. (871° C.), cooled in air.
Held 5 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	52 500
Tensile strength, lb. per sq. in.	91 700
Elongation in 2 in., per cent	25.5
Reduction of area, per cent	54.1

FIG. 11.—Structure Near Surface, Main Rod No. 899, Normalized. Etching Reagent, 2-Per-Cent Nital (× 100).



Treatment

Held 5 hrs. at 1600° F. (871° C.), cooled in air.
Held 5 hrs. at 1200° F. (649° C.), cooled in air.

Tensile Properties

Yield point, lb. per sq. in.	53 900
Tensile strength, lb. per sq. in.	88 300
Elongation in 2 in., per cent	25.0
Reduction of area, per cent	56.0

FIG. 12.—Structure in Center, Main Rod No. 899, Normalized. Etching Reagent, 2-Per-Cent Nital (× 100).

the heater. Cold ingots should not be placed in a hot pit, but time must be allowed for the ingot to become solid throughout before it is placed in the pit. The temperature of the pit should be uniform and the direction of the flame frequently reversed. The usual practice is to reverse the flame every 15 minutes. If a comparatively cold ingot is placed in a very hot pit, the outside of the ingot will expand and pull away from the interior section and many times these bursts are persistent and will not weld in the subsequent rolling. Ingots should therefore be heated slowly to the rolling temperature, and after reaching this temperature should be held a sufficient length of time to insure a uniform temperature throughout. This temperature should not be less than 2100° F. (1149° C.) for the smaller sizes and 2200° F. (1004° C.) for the larger which require longer time in rolling.

The results of improper rolling are checks and strains. The temperature of the ingot at the time rolling is started should be not less than 2050° F. (1121° C.) for the small sizes and 2150° F. (1177° C.) for the large. Burnt edges show up very quickly in the rolls, and internal checks may break through to the outside. In order to prevent strains, the reduction in each pass should not exceed 2 in., and the reduction from the ingot to the finished bloom should be 4 to 1. The discard should be at least 25 per cent of the ingot, and more if necessary, as no hard and fast rule may be made for this important operation. Discard should be continued until no evidence of pipe can be seen. Pipe, if not discovered while the ingot is being forged, or before the forging leaves the shop, will cause failure of the forging after it is placed in service by starting a progressive fracture. Segregations of impurities are indications of pipe in many cases, and should be carefully watched. In order to prevent the formation of pipe, the use of the Gathmann molds and hot tops is recommended, and has been a great factor in making sound ingots.

In the process of forging it is very important that the billets be carefully heated to the forging temperature. In the winter months the blooms or billets become very cold in shipping, and they should be placed inside the forge shop in a warm place for at least 24 hours before they are placed in the furnace. The best practice is to use a preheating furnace into which the billets are charged at room temperature, and which is then slowly heated to not over 1000° F. (538° C.) at a rate of not over 150° F. (83° C.) per hour. This insures freedom from internal checks due to rapid heating. The billets are then removed from this preheating furnace and placed in the forge furnace where they are raised to the forging temperature. The same precautions are necessary here as were recommended for the soaking pit in the steel mill. The regulation of the fuel and air should be such as to obtain the least amount of deposit on the billets while heating, as this insures more efficiency from the heat of the furnace and lessens the chance of overheating. Billets should be soaked at the forging heat a sufficient length of time to insure complete penetration and absolute uniformity of temperature. Sharp flames must not be allowed to come in contact with the edges of the billets or checks will be produced due to overheating, and there must be no local heating in the forge furnace. The temperature of the bloom or billet must not be greater than 2100° F. (1149° C.).

There are no standard rules which may be set for the working of the bloom under the hammer, but a hammer of sufficient size to insure working to the center of the bloom or billet must be used. In working a large bloom if the penetration is sufficient, the forging will have a tendency to bulge in the center and on the end. If the working does not penetrate to the center of the bloom, the forging will be concave in the center and on the end. Too light a hammer results in very unsatisfactory physical results, and a test taken near the center of the forging will not meet the physical requirements specified. Microscopic examination readily establishes this condition. If the temperature is too low for proper forging, there will be no penetration of sufficient depth, and the resulting forging will show evidences of cold work. Cold-worked forgings are in a strained condition which is not readily relieved in subsequent normal heating. The working of the forging must stop when the steel ceases to work freely under the hammer, and no definite temperature can be given for this point, as scale forms so quickly on the forging that the temperature cannot be taken with the optical pyrometer. It would be uselsss in any case, as the temperature of the surface may be higher or lower than that of the interior, if the heating has not been properly done.

Cooling after forging is a very important process, and all carbon-vanadium forgings should be slowly cooled in a bed of ashes. This considerably decreases the likelihood of strains being set up or the formation of cooling cracks due to hammer strains.

Checks can be produced in the normalizing process by too rapid heating, for the forgings are usually cold when placed in the normalizing furnace, and too rapid heating may cause surface expansion, causing the outside of the forging to pull away from the interior cold section. The rate of heat application should be maintained constant, and after the proper temperature has been reached, should be held for at least one hour per inch of diameter or thickness of the largest section of the forgings being treated. Forgings should be supported several inches above the surface of the car to insure complete circulation of the hot gases to every part of the forgings. In the case of large forgings such as crank-pins, axles, or the large heads of rods it is advisable to have these rough-bored so that the soaking and subsequent cooling action of the air will have the desired normalizing effect. The normalizing furnace should be so constructed that no flame can touch the forgings, and an adequate number of thermocouples should be used to enable every part of the heating chamber to be maintained at the same temperature.

In conclusion, it may be said that there are only two kinds of steel, "normal" and "abnormal" or good and poor. If a steel is normal, that is, if it has been properly made under right conditions and with the practice outlined herein as good mill practice, and has then been properly forged, the problem of normalizing to meet the usual specifications for this class of material presents no difficulties whatever and normalizing temperatures are not critical. The material will then readily meet the specifications of the various railroad companies for this class of material. Furthermore, these forgings will withstand the severe service demanded of them in steam locomotive operation. A series of photomicrographs are given to show the various conditions outlined in this paper.

STRENGTH OF WELDED JOINTS

BY J. R. DAWSON[1]

SYNOPSIS

The investigations described in this paper were carried out to secure information about the physical properties of oxy-acetylene welds and to determine the extent of uniformity and dependability that could be secured in the welded joint.

Tension test results are included for 78 specimens prepared by one welder and for single welds made by a large number of welders. The welds were made in firebox steel and in higher strength steel plates, using welding rods of improved quality.

A procedure control is outlined which is suggested as a means of securing dependable results in structures fabricated by welding.

INTRODUCTION

The rapid increase in the application of fusion welding in the preparation of steel structures makes it highly desirable that complete data should be available about the physical properties of steel welds as now made. It is well known that high-quality welds can be obtained and it was one of the purposes of the work described herein to investigate the uniformity of the quality of welds to determine the dependability of this method of joining steel. For this purpose two lots of welds were made. In one case, 78 oxy-acetylene welded specimens were prepared on successive days by one welder and in the other, tests were made of a large number of oxy-acetylene welds, each one prepared by a different workman.

TESTS OF WELDS PREPARED BY SINGLE WELDER

The first set of tests was carried out in a booth which was a part of the International Steel Exposition held in connection with the 1924 annual meeting of the American Society for Steel Treating at Boston. The welds were made under rather trying conditions due to interruptions by the visitors at the exposition and the consequent interference with the steady progress of work.

Material.—For this part of the investigation, 6 by 9-in. firebox quality steel plates $\frac{3}{8}$ in. thick were joined by the oxy-acetylene method by a welder of good but not exceptional ability in this variety of welding. The steel plates ranged in tensile strength from 52,000 to 58,000 lb. per sq. in. The edges to be joined were beveled on both sides of the plate so that the total angle to be filled in on each side was 90 deg. The layout of these plates is illustrated in Fig. 1.

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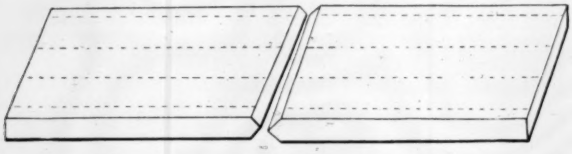

FIG. 1.—Layout of Plates to be Welded.

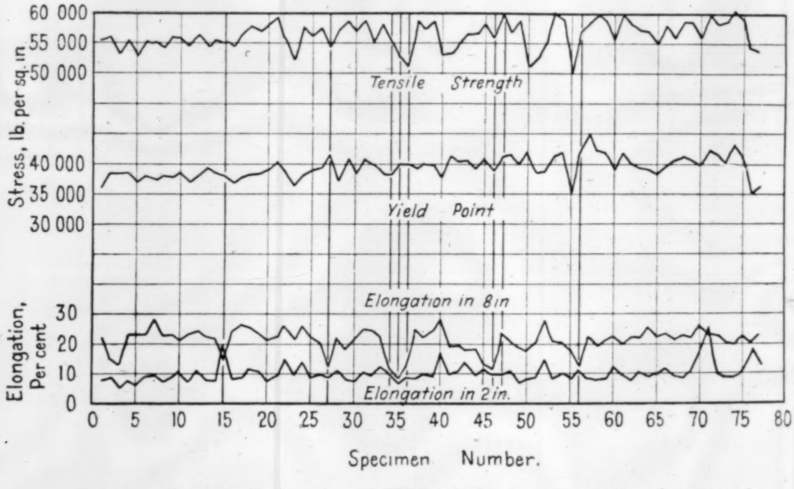

FIG. 2.—Tensile Properties of Welded Firebox Steel Specimens.

Specimens in groups of three are for the same plate, e.g., numbers 1, 2 and 3 are from one plate, numbers 4, 5 and 6 from another, etc.

Specimens that fractured in the weld are indicated by the heavy ordinate lines.

Welding and Preparation of Specimens.—The welding rods used were a special commercial type of the following average composition: carbon, 0.17 per cent; manganese, 0.82 per cent; silicon, 0.53 per cent. Plates were welded first on one side and then on the other. After each welded plate had cooled, it was clamped in an oxy-acetylene straight-line cutting machine, and three strips $1\frac{1}{2}$ in. wide and 18 in. long with the weld in the center were cut out as shown by the dotted lines in Fig. 1. The edges of these strips were ground smooth and enough grinding was done at the weld so that the width of the weld was less than at any other point on the strip. The top and bottom of the weld were ground flush with the remainder of the specimen, so that the bar was substantially smooth on all four sides, there being no reinforcement whatever at the joint.

Tension Tests.—Seventy-eight of these strips were broken in the 50,000-lb. tension testing machine loaned by the Tinius Olsen Co. In Fig. 2 are graphs showing the details of the tensile strength, yield point as obtained by the drop of the beam, and percentage of elongation in 8 in. and in 2 in.

The fracture of 70 of the 78 specimens tested occurred entirely outside the weld at a distance of from 1 to 4 in. For those that failed in the weld, the tensile strength developed was of the same order as for those that broke in the plate metal. In the following specimens the fracture was in the weld: Nos. 15, 27, 34, 35, 36, 46, 47 and 56. These may be located on the heavy ordinates in Fig. 2. Their average tensile strength was 54,900 lb. per sq. in. As can be noticed by reference to Fig. 2, the location of the fracture has considerable effect on the percentage of elongation. The elongation in 2 in. was measured at the weld regardless of the location of the fracture. Because the weld was ground until the area of the section through the weld was smaller than at any other point of the specimen, it is clear that the joint was the strongest part of the welded firebox steel. Since the specimens were prepared by oxy-acetylene cutting, followed by grinding, there were necessarily irregularities which caused slight errors in measurement and, except for this fact, the variation in the values for tensile strength and yield point would have been found even less than are shown in the graphs.

TABLE I.—REDUCTION OF AREA, SPECIMEN NO. 53.

Tensile strength, 55,500 lb. per sq. in.
Elongation, 15 per cent in 2 in.; 27.5 per cent in 8 in.

Location	Measurements Before Testing, in.	Measurements After Testing, in.	Areas Before Test, sq. in.	Areas After Test, sq. in.	Reduction of Area, per cent
3 in. from weld	1.435 by 0.377	1.336 by 0.347	0.541	0.463	14.4
2 in. from weld	1.435 " 0.375	1.293 " 0.335	0.538	0.434	19.3
Adjacent to weld	1.400 " 0.360	1.315 " 0.335	0.504	0.441	12.5
Center of weld	1.379 " 0.375	1.345 " 0.369	0.517	0.497	3.9
Adjacent to weld	1.392 " 0.375	1.288 " 0.340	0.522	0.438	16.1
2 in. from weld	1.445 " 0.388	1.040 " 0.220	0.560	0.229	59.1
3 in. from weld	1.455 " 0.388	1.345 " 0.350	0.569	0.471	17.2

In Table I is a typical example of the measurements taken and results obtained in testing one of these welds. The area of the weld was smaller than at any other point in the specimen, and after breaking, the section of greatest area was at the weld. If fracture had occurred inside the 2-in. gage marks the value for elongation in 2 in. would have been much greater.

This table is included to aid in showing that values for elongation obtained in tension tests of welded specimens should not be expected to compare with those for the usual unwelded specimens. The properly made oxy-acetylene weld obtained with the better quality welding rods is stronger and stiffer and of higher yield point than the usual low-carbon steel plate. For this reason, regardless of the nature of the strain applied, very little of the deformation that occurs is in the weld. When the break is outside the weld and a measurement is taken in the 2 in. over the weld, the elongation that could be secured in this portion of the specimen is not even approached and furthermore, only a portion of the 2-in. length consists of weld metal. In the 8-in. measurement, the weld which has not been elongated is included and for this reason that measurement also is not comparable with the measurement in a steel that does not contain a weld.

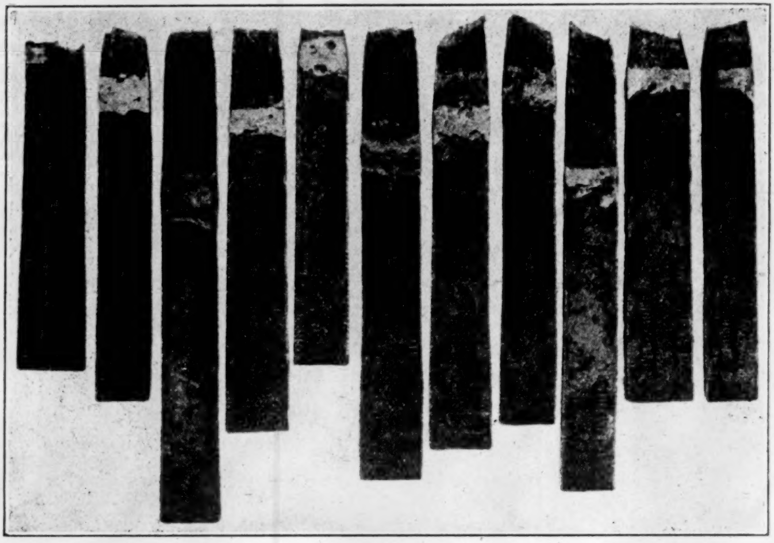

FIG. 3.—Appearance of Fractured Specimens.

In the standard test specimen, instead of a uniform 8-in. length with shoulders at the end, the higher strength weld produces the same effect as would a heavier section of unwelded material at the center, and, therefore, the 8-in. length corresponds to two 3-in. sections that are subject to deformation with a shoulder about 2 in. long between them. It is obviously misleading to measure the elongation in 8 in. when practically all the stretching occurs in about 6 in. of the length. Since the fracture is usually not included in the 2-in. measurement over the weld, these same conditions apply to an even greater extent in their effect on this elongation value.

Fractured specimens as selected at random are shown in Fig. 3. The welds are at the light areas on the specimens where the reinforcement was ground off. Even though the edges were ground to make the width least at the welds, the deformation that occurred was in the plate and it can be noticed that in the tested specimens, there was sufficient necking-down on both sides of the weld so that its section is greater than that of the base metal. The specimen on the extreme left broke through the weld, and in the others, failure occurred at various distances from the weld.

TABLE II.—TESTS OF MACHINED SPECIMENS.

Specimen	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Elongation in 8 in., per cent	Fracture
No. 1	34 500	53 000	11.0	22.5	Outside weld.
No. 2	34 500	53 400	9.0	23.0	Outside weld.
No. 3	34 250	52 800	11.5	21.0	Outside weld.
No. 4	33 700	51 200	30.0	20.0	Outside weld.
No. 5	35 050	53 250	7.0	8.1	In weld.

Five welds made at the same time as those just described were machined all over to the standard A.S.T.M. 8-in. test specimen. The results obtained in pulling these are recorded in Table II. Specimen No. 5 broke through the weld at a strength nearly equal to the average of the eight plotted in Fig. 2 that also broke in the weld, and the other four failed in the base metal at various distances from the weld. The fracture in No. 4 was outside the weld but within the 2-in. gage marks and for this reason the percentage of elongation in 2 in. was high.

The work that has been described was duplicated at the International Steel Exposition held in Cleveland in 1925. Fifty-one specimens were broken and the fracture of 46 of them was outside the weld. The results covered the same range as is shown in the graphs of Fig. 2.

TESTS OF WELDS PREPARED BY VARIOUS WELDERS

Oxy-acetylene welds in $\frac{3}{8}$-in. steel plate made by a wide variety of operators but all using the improved grade of welding rods were secured. The strength of the firebox plate ranged from 52,000 to 58,000 lb. per sq. in. The average strength of the unreinforced specimens made by a group consisting of 63 welders was 52,800 lb. per sq. in. The results obtained in testing these specimens fell within the following groupings:

2 specimens	35 000 to 40 000 lb. per sq. in.
2 specimens	40 000 to 45 000 " " " "
9 specimens	45 000 to 50 000 " " " "
23 specimens	50 000 to 55 000 " " " "
27 specimens	55 000 to 60 000 " " " "

Of another group of 14 welds, each made by a different operator, the average strength was 62,400 lb. per sq. in. and the average elongation in 2 in. was 14 per cent. The range of tensile strength values was: minimum, 52,500 lb. per sq. in.; maximum, 68,400 lb. per sq. in. In five of these specimens the fracture was through the weld and in the remainder, it took place from 2 to 4 in. outside the weld. The reason the latter group had higher average strength was that the base metal in which the welds were made was of high strength, being 63,400 lb. per sq. in. Many of the welds in the group that averaged 52,800 lb. per sq. in. tensile strength also broke outside the weld so that the stress the weld was capable of withstanding was not developed. No selection of welders was made to prepare these coupons and therefore the welds represented the work of average shop welders where there is always change due to men entering and leaving the employment. In each case, the entire group made welds so that the work of the poorest as well as of the best workman was included.

It had been suggested that, although under static loading, weld test results might be of a favorable nature, perhaps under shock or bending stresses they would not be so favorable. To determine the effect of shock, seven welds of the same size as those already described were placed in the tension testing machine and when the yield point had been passed, the specimens were struck at the weld a blow of 16 ft-lb. These blows were continued as the load was increased up to the time that failure occurred. There were 36 blows for each specimen; three blows which were severe enough to slightly bend the specimens were struck after each of twelve approximately uniform increments in load. The results obtained were apparently entirely unaffected by the shock received, for the breaks were outside the welds and the tensile values corresponded with those for the unwelded firebox steel. To determine the effect of bending a specimen, two $\frac{3}{8}$-in. plates were welded together so that the angle between the plane of one and an extension of the plane of the other was 30 deg. They were then placed on the bed of the testing machine and loaded at the welds until they flattened out. The welds were of sufficient strength and stiffness that bending was confined to the firebox steel base metal. In a large number of tests with the rods and base metal referred to, the results obtained were unfailingly the same. An account of these tests is included to show that tensile strength results are a reliable indication of the quality of welds made in steel plate.

TABLE III.—OXY-ACETYLENE WELDS IN HIGH-STRENGTH PLATE.

Specimen	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Fracture
No. 1	49 500[a]	66 800[a]	18.0[a]	$\frac{1}{4}$ in. from weld.
No. 2	45 600[a]	64 100[a]	12.5[a]	3 in. from weld.
No. 3	52 000	71 200	21.0	Part plate, part weld.
No. 4	49 800	68 900	13.0	No visible defects.
No. 5	51 700	69 200	13.0	No visible defects.
No. 6	54 500	69 500	13.0	No visible defects.
No. 7	56 900	64 500	9.0	No visible defects.
No. 8	52 700	64 900	16.0	No visible defects.
No. 9	53 000	67 700	10.0	No visible defects.
No. 10	......	53 300	3.0	Slight poor fusion.
No. 11	59 500[a]	76 400[a]	9.0[a]	1 in. from weld.
No. 12	57 700	71 800	10.0	No visible defects.
No. 13	53 300	69 600	13.0	No visible defects.
Average	53 500	67 000	12.1	

[a] These values are not included in the average because the weld metal was not fractured and the results do not represent true weld-metal properties.

TENSILE PROPERTIES OF WELD METAL

It will be noted that the tests so far recorded have done little more than record the minimum strength of a double-V weld made with a high test welding rod. In other words, nearly all the specimens broke in the plate rather than in the joint. To determine the tensile properties of the weld metal itself, welds were made in high-strength plate with the rods used in the tests already described. Thirteen welders employed in the same shop each prepared a specimen. The results obtained in tests of these welds, which were not reinforced, are given in Table III.

SUMMARY

Tests in high-strength steel plate indicate that the tensile properties of the weld metal, deposited from a rod of the high-manganese, high-silicon steel composition described, under the oxy-acetylene flame by experienced workmen may be conservatively stated as: tensile strength, 65,000 lb. per sq. in.; yield point, 50,000 lb. per sq. in.

Tests in firebox steel having strength of 52,000 to 58,000 lb. per sq. in. show that a double-V weld made with the above rod will develop the full strength of the plate, even without reinforcement at the weld. This can be done time after time by any properly trained welder.

TABLE IV.—PROCEDURE CONTROL FOR WELDING, AND COMPARISON TO SHOW THAT ITS REQUIREMENTS ARE SIMILAR TO THOSE FOR RIVETING AND REINFORCED CONCRETE.

	Welding	Reinforced-Concrete Construction	Riveting
A	Determine whether welding operators have the requisite knowledge and ability to do the type of welding under consideration.	Determine whether those doing the work know how to prepare the forms, and mix the concrete.	Determine whether those doing the riveting have been properly trained in the work.
B	Select materials of good quality.	Select materials of good quality.	Select materials of good quality.
C	Design the joints correctly.	Design the structure correctly.	Design the structure correctly.
D	Make suitable preparation of the joints for welding.	Have foundation in suitable condition to support the structure, reinforcing properly located, and the forms correctly and securely placed.	See that fitting up is properly done.
E	Apply approved technique.	Pour concrete according to approved practices.	Drive rivets according to approved practices.
F	See that sufficient inspections and tests are carried out during construction and at the completion of the work.	See that sufficient inspections and tests are carried out during construction and at the completion of the work.	See that sufficient inspections and tests are carried out during construction and at the completion of the work.

PROCEDURE CONTROL

The results obtained in this investigation of the strength of welded joints justify confidence in any similarly prepared welds, and yet the claim may rightly be made that welds sometimes fail. Although it is a fact that riveted or reinforced-concrete structures have also failed, these two methods of construction deservedly hold the confidence of engineers and the reason is that fundamentally correct standards of testing and procedure have been worked out and are universally followed.

As compared with these methods of construction, fusion welding is a recent development, but the same sort of control is being applied to it as to riveting or to concrete construction. Where failure has occurred in any of them, it has been due to neglect of some of the fundamental requirements set down in the following comparison of the procedure that must be followed if success is to be obtained. Experience has demonstrated that where this "procedure control" is observed, fusion welding is deserving of the same confidence that is accorded the other methods of construction.

For example, good quality cement, sharp sand free from clay, and properly sized coarse aggregate are required to make good concrete. Structural steel and rivet steel must meet certain standards as to inspection and tests. In the same way, to secure best results, the welding rods must be of correct composition, free from excessive quantities of impurities and of good flowing qualities under the torch. These conditions apply also to the metal to be welded. A similar comparison could well be made for each of the items set down in Table IV.

In this paper the intent has been to include data that may be of use to engineers when information about the physical properties of fusion welds is required. Where there is knowledge of the properties of welds and suitable standards of control are employed, confidence in this method of producing a continuous metal structure rather than a jointed one is well justified.

DISCUSSION

Mr. Moore. MR. H. F. MOORE.[1]—In view of the rather small amount of test data available on the strength of welded joints under repeated stress (fatigue strength), and of the fact that such data as are available indicate that welded joints are relatively less strong under repeated stress than they are under static stress, I should like to ask the author if he has any test data on the fatigue strength of autogenous welded joints. I should also like to ask him whether he has any data on the strength of thick plates autogenously welded.

These questions are asked, not with any intention to discredit autogenous welded joints in service, but with the hope that they will bring out more information on the behavior of welded joints under repeated stress, and will bring out any improvements which have been made in the technique of welding thick plates.

Mr. Thum. MR. E. E. THUM.[2]—I have some additional information on the strength and uniformity of welded joints when made by welders selected with reasonable care. This is drawn from a series of tests made on operators employed by the Oxweld Railroad Service Co., the tests being seventy-five or eighty in number, extending over a period of six years. For the first three years of that period, the rod used was one sold commercially as Norway iron, but which was merely a first-class low-carbon steel. During the last three years a rod such as described by Mr. Dawson and known commercially as "high-test rod" (0.20 per cent carbon, high in silicon and manganese) was used by them. In plotting these tests, of which there were about 500, those which broke in the plate were segregated from those which broke in the weld, and were then plotted in order of decreasing magnitude. Horizontal ordinates represent in the accompanying Fig. 1 the cumulative percentage of the total. The early three years on Norway iron showed that only about 11 per cent broke in the plate, which was supposed to be of good quality. The plate was taken from railroad stock all over the United States, and consequently varied somewhat. This welding, by the way, was single-V welding in about $\frac{3}{8}$-in. plate, with excess weld metal ground flush to the plate. However, the breaks which occurred in Norway iron (which is not

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Manager, Technical Publicity Department, Linde Air Products Co., New York City

Mr. Thum.

nearly the best welding rod available to-day) varied from 57,000 down to 30,000 lb. per sq. in.

Some interesting things about "high-test rod" are shown on these curves. One is the enormous advantage to be had by using welding materials of very high quality. It was found immediately upon using this welding rod that only the best of flange steel should be used for test metal, but, even so, nearly half the specimens broke in the plate rather than in the weld. Another interesting thing is that men of all grades of ability are able to increase the quality of their work by some 11,000 lb. per sq. in. by merely using a better welding rod. Since the poorest man in the shop is the weakest link, as it were, this is a most important consideration.

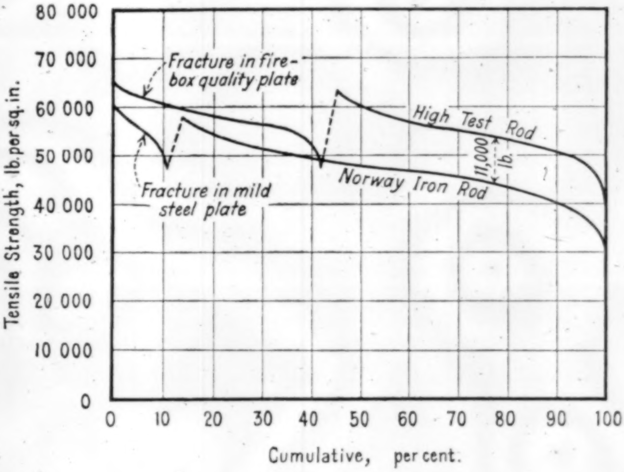

FIG. 1.—Results of Tests.

Furthermore, note the good workmanship of men who have been selected with a proper regard to their qualifications. Such statements as these are frequently made: "Welding depends entirely upon the welder"; "There is no way of telling when you have a good workman." Here, I think, are some definite figures which prove the error of such views. With high-test rods, single-V welds, ground flush, 95 per cent of these men selected with due regard to their ability were able to make an oxy-acetylene weld as good as or better than the firebox quality steel delivered under specification to the American railroads, and with no greater variation in physical strength, maximum to minimum.

Mr. Thum. In order to appraise this mass of data and establish figures for design purposes, the data given in Fig. 1 were plotted in the form of a probability curve, Fig. 2. (The strength of joint in those pieces which broke the plate was assumed to be 5000 lb. per sq. in. greater than the strength of the plate.) From Fig. 2 it may be deduced that the expected strength of single-V welds made by the oxy-acetylene process, all reinforcement ground off, is as follows:

Using Norway iron welding rod................	47 000 lb. per sq. in.
Using "high-test" steel rod....................	58 000 " " " "

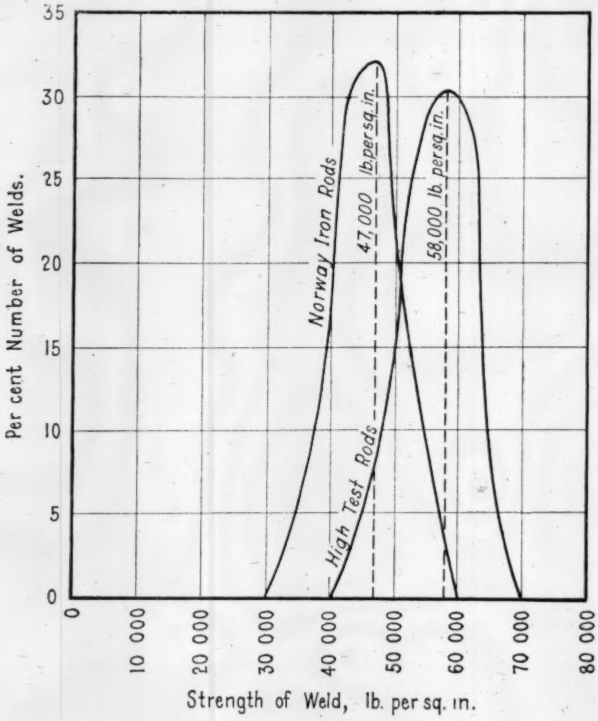

FIG. 2.—Most Probable Average Ability of Welders.

These figures represent, with a high degree of probability, the average work to be expected by welders working under what may be termed "procedure control," and designers can use them with confidence.

The factor of safety selected for each individual design would take into consideration the poorest joint in the lot; but for either kind of welding rod, this is well above the elastic limit of low-carbon steel suitable for welding.

Mr. Dawson.

MR. J. R. DAWSON.—Mr. Thum has already partly answered Mr. Moore's question. Our past experience in welding in this country was largely with rods that do not produce as good results as can be obtained at this time. The other point is that the weld is usually a small part of the structure and is generally reinforced sufficiently for the purposes to which the structure is to be put. Now if you subject a weld, as it is reinforced, to fatigue tests, the weld is stronger and stiffer than the metal on either side of it. For that reason the failure would be expected to occur, not in the weld, but in the base metal. It might be of interest to Mr. Moore and others to consider the action of a riveted joint under fatigue tests.

In reply to Mr. Moore's question with regard to the making of welds in thicker materials, I would say that usually welders are accustomed to working with the thinner materials and are not as well experienced or as much practiced in handling the heavier sections, except in cast iron welding. For that reason an occasional heavy weld is likely to contain defects due to lack of practice. It is also true that grain growth and the final arrangement of non-metallic materials in a heated zone depends upon the temperature attained and the length of time the material is held at the temperature. When a heavier weld is made, the high temperature is maintained for a longer time and for that reason the weld in the thicker plates is somewhat less efficient. It should be also remembered that in reasonably good quality low-carbon steel, where most welds are made, the temperature effects are not at all to be compared with those resulting from similar heating of higher carbon materials.

THE MAGNETIC ANALYSIS OF HIGH-SPEED STEEL

By Thomas Spooner[1]

Synopsis

There are presented here the results of an investigation of the correlation between the magnetic and electrical properties and the heat treatment of a series of bars of high-speed steel. A range of quenching temperatures from approximately 2100 to 2400° F. (1149 to 1316° C.) and drawing temperatures to 1150° F. (621° C.) are covered. Various methods of test are considered. The most suitable ones so far investigated involve an alternating current magnetic test at fairly high magnetizing forces. Such a test, in general, gives a very quick and reliable indication of the uniformity and absolute values of heat-treating temperatures, and discloses differences which are not brought out by the usual methods of examination.

Some data are also given concerning the effect of time at the high temperatures and the effect of different quenching methods. Finally, magnetic data are presented showing the aging effects observed after heat treatment. The progressive changes at room temperature are comparatively large and doubtless of considerable practical significance.

Introduction

The main purposes of this investigation were to determine the relation between the electrical and magnetic properties and heat treatment of high-speed steel with the object of devising a simple inspection method for heat-treatment control, and to obtain fundamental data for the purposes of magnetic analysis. This work was done under the auspices of Committee A-8 on Magnetic Analysis of the American Society for Testing Materials. Two previous investigations on high-speed twist drills in cooperation with the Cleveland Twist Drill Co.[2] (the second investigation is still under way) showed so many variations that it was considered advisable to obtain fundamental data on simpler specimens under conditions of known and completely controlled heat treatment, the magnetic and electrical properties to be determined at each stage of the heat treatment.

This paper may be considered as a report of progress. Numerous other tests on this same material have been and are being made by other members of Committee A-8. It is hoped that these tests will further assist in developing practical inspection and test methods.

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[1] Research Engineer, Westinghouse Research Laboratory, East Pittsburgh, Pa.

[2] W. B. Kouwenhoven, "Magnetic Tests of A.S.T.M. Drills," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 635 (1924).

SAMPLES

One hundred and fifty pounds of ½-in. square high-speed steel bars were obtained from the Horne Steel Products Co. These bars were supposed to be all from the same heat. Each bar was assigned a letter and then cut to approximately 10-in. lengths, giving from 10 to 15 samples per bar. Each 10-in. sample was given a number and the numbers were in the order in which the samples were cut from the original bar. The marking of some of the bars was undecipherable or was omitted accidentally. In these cases the bars were marked X. The specimens were then ground to 0.45 in. (1.14 cm.) square. After machining the bars were normalized at 1650° F. (899° C.).

Before further heat treatment, the specimens were tested magnetically in a number of ways to determine their uniformity. In subsequent work the abnormal bars were excluded. It was found that one set of samples (marked K), all from one of the original bars, were quite different from the rest as indicated by certain tests. A chemical analysis revealed the cause of the difference:

	Carbon, per cent	Silicon, per cent	Sulfur, per cent	Phosphorus, per cent	Manganese, per cent	Nickel, per cent	Chromium, per cent	Vanadium, per cent	Tungsten, per cent
Normal Material..	0.79	0.42	0.042	0.031	0.176	0.10	3.42	0.88	17.35
K Sample.........	0.70	0.30	0.030	0.021	0.173	0.11	3.97	1.49	14.03

HEAT TREATMENT

The first heat treatment after normalizing consisted of a quench carried out on 75 bars, 15 for each temperature, as follows:

The samples were pre-heated in a Bellis carbon-steel-quenching salt bath. The mean temperature was 1650° F. (899° C.) with a plus or minus variation of perhaps 30° F. (17° C.).

The samples were heated for quenching in a Bellis high-speed salt bath in a mild-carbon-steel pot made from a piece of shafting. The heating was by means of a high-frequency induction furnace taking about 150 amperes at 15,000 cycles. The temperature was read by means of a Leeds & Northrup optical pyrometer sighted on the bottom of a closed-end iron tube immersed in the bath. The temperature control was manual and the optical pyrometer readings were held to ±5° F. (3° C.). The optical pyrometer was calibrated by comparison with a platinum - platinum rhodium couple standardized by the U. S. Bureau of Standards. The pyrometer was later checked at Nela Park. The specimens were heated for 3 minutes in the quenching bath at various temperatures, namely, 2109, 2211, 2316, 2366 and 2419° F. (1154, 1211, 1269, 1296 and 1326° C.).

The quenching was performed in two steps. The first quenching was in a salt bath held at a mean temperature of 1250° F. (677° C.), with a plus or minus variation of about 30° F. (17° C.), the control being automatic. After 2 minutes, the samples were transferred to an oil bath at room temperature.

The heat treating was done under the direction of Mr. P. H. Brace.

TEST METHODS

Fundamentals of Alternating Current Magnetic Tests.—Fig. 1 (*a*) illustrates one of the simplest forms of magnetic test using alternating current. If the principle of this test is understood, the operation of the other alternating-current tests will be clear. Alternating current from any convenient source energizes the primary winding N_1 through a resistance R_1. A secondary winding N_2 is connected to some convenient voltage-measuring device such as an a.c. galvanometer or a.c. potentiometer. These two windings may be in the form of a straight solenoid in which the sample is placed. The galvanometer G or a potentiometer may be connected either to the secondary winding N_2 or across the primary resistance R by means of the switch Sw.

Now referring to Fig. 1 (*b*), assume that it is desired to measure two components of exciting current, one proportional to the permeability of the sample and one proportional to the loss. Assume also that E, the voltage induced in N_2 and therefore the flux in the sample, is kept constant by adjusting the voltage applied to N_1. Now, with no sample in the coils, the exciting current I would be 90 deg. out of phase with E, and in phase with the flux ϕ. Since, however, the sample has certain losses (hysteresis and eddy current), I will be shifted toward E by an amount depending on the magnitude of the losses. If the field of the galvanometer G is so adjusted in phase that it reads a maximum when connected to N_2 and is then connected across R, its reading will be proportional to the loss component I_W of exciting current. If the phase angle of the field is shifted 90 deg., the galvanometer reading will be proportional to the magnetizing component I_M of the exciting current, that is, the reading will be inversely proportional to the permeability of the core material for a definite voltage induced in N_2.

Now suppose as another test, Fig. 1 (*c*), that there is a high resistance in series with N_1 and that the primary current I is kept constant. The galvanometer G is connected to N_2 and the phase angle of its field adjusted until with no sample in the coils it reads a maximum. Under these conditions the flux ϕ is in phase with I and the measured E is 90 deg. out of phase. If now a sample is introduced into the coils, the reading of E is greatly increased due to the larger flux and there is a phase shift of E and ϕ due to the losses in the sample. The reading of G will now be proportional to E_B or to the permeability of the sample. If the field of E is shifted 90 deg., the reading of G will be proportional to E_W or the loss in the sample.

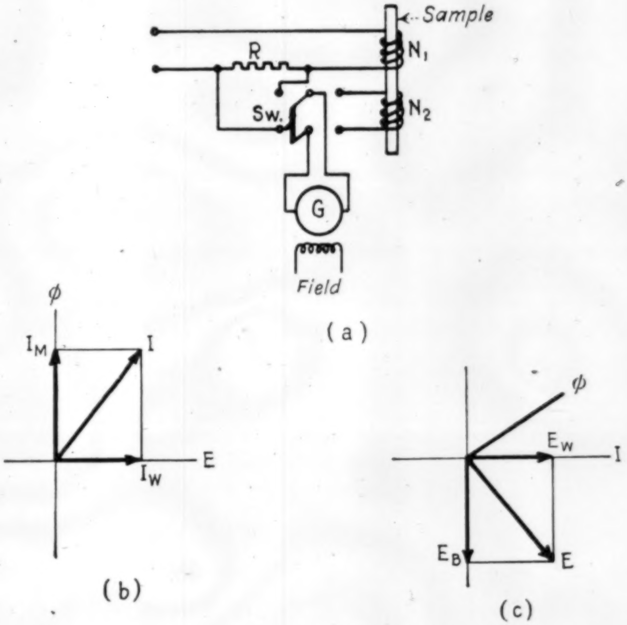

FIG. 1.—Simple Alternating-Current Magnetic Tests.

Sometimes when a differential method, to be described later, is used with a standard sample, the galvanometer may be set for a maximum or for a zero reading when connected across the secondary coil on the standard sample. In this case the galvanometer reading indicates the difference in induction ΔE_B for the two samples or the difference in loss ΔE_W.

In the case of constant exciting current, Fig. 1 (*c*), the loss figure E_W is a function not only of the loss in the sample but also of the permeability, since if the permeability is high the induction will be high, thus tending to increase the losses. For this reason if a segregation of permeability and losses is desired the method of Fig. 1 (*b*) is preferable. The de Forest[1] test method usually measures values which are a function of both E_B and E_W (or ΔE_B and ΔE_W).

Comparison of A.C. and D.C. Methods of Test.—Alternating-current methods of test are often preferable to direct current since an ordinary commercial lighting circuit may be used as a source of power and the measuring instruments may be more rugged, portable and more easily read. Alternating-current methods are also much quicker if loss values are desired and necessary if eddy-current losses are to be measured. Direct current measurements are preferable if results are required at high inductions; or if the samples have such large cross-sections that the eddy currents become excessive. Under the last condition, a.c. tests will give results only for the surface portion of the material and will produce excessive heating and therefore temperature effects.

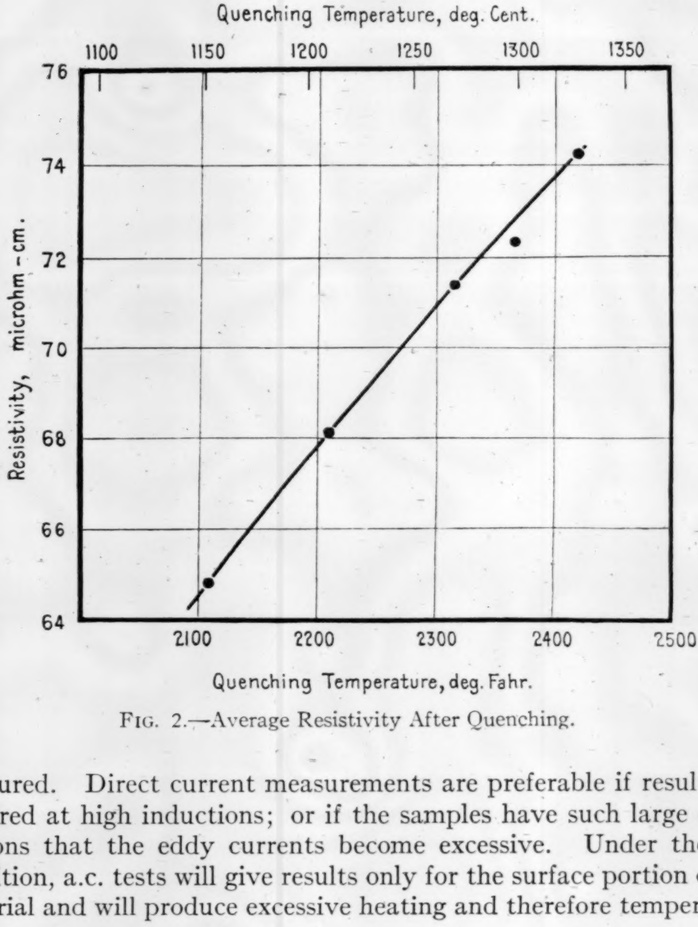

FIG. 2.—Average Resistivity After Quenching.

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[1] A. V. de Forest, "A New Method of Magnetic Inspection," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II., p. 611 (1923).

TEST RESULTS AFTER QUENCHING

Magnetic and Electrical Tests: Six types of test were applied to the samples after quenching: namely,

1. de Forest magnetic test;[1]
2. Resistivity;
3. A.c. potentiometer magnetic test;

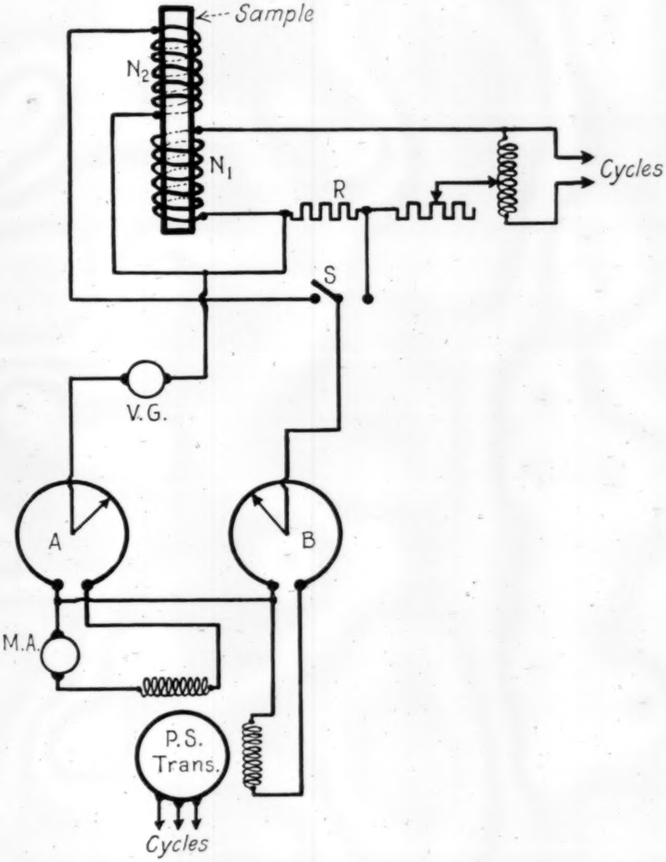

FIG. 3.—Diagram of Connections for Solenoid Test with A.C. Potentiometer.

4. Ballistic magnetic test;
5. Fahy simplex (d.c. and a.c.) magnetic test;
6. A.c. differential magnetic test.

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[1] A. V. de Forest, "A New Method of Magnetic Inspection," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 611 (1923).

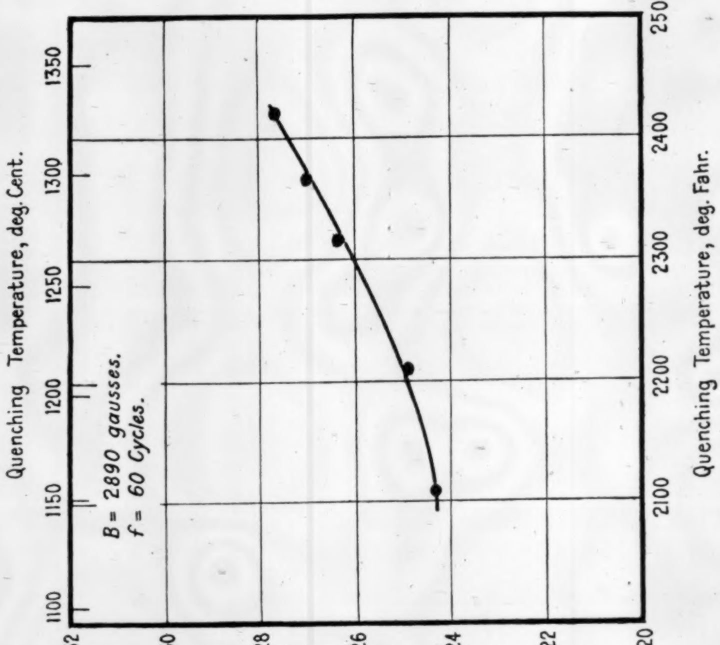

FIG. 4.—Average Loss Currents for Solenoid Test.

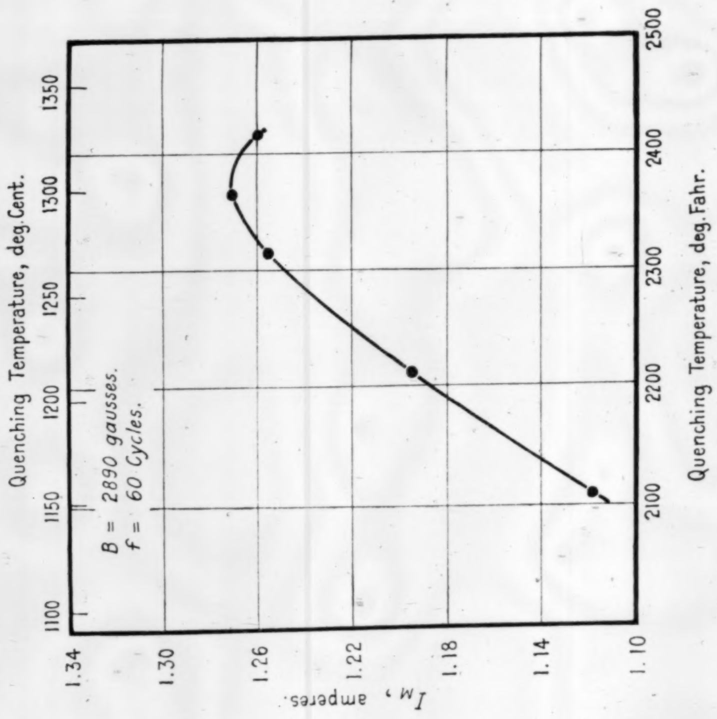

FIG. 5.—Average Magnetizing Currents for Solenoid Test.

de Forest Magnetic Test.—The de Forest test was made using 60 cycles and a short solenoid applying a field of 345 ampere turns. The three sets of bars having the three lowest quenching temperatures were well separated, but the two sets having the highest quenching temperatures could not be distinguished by this test. Since then, however, Mr. de Forest has obtained a better separation by a more suitable choice of coordinates.

Resistivity Test.—The resistivity tests were made by means of a Wolf potentiometer and standard resistance. The average results are given in Fig. 2. There is no difficulty in making resistivity tests on freshly-machined bars, but after heat treatment it is necessary to remove the high-resistance scale before satisfactory electrical contact can be made. This is sometimes rather difficult and always requires time.

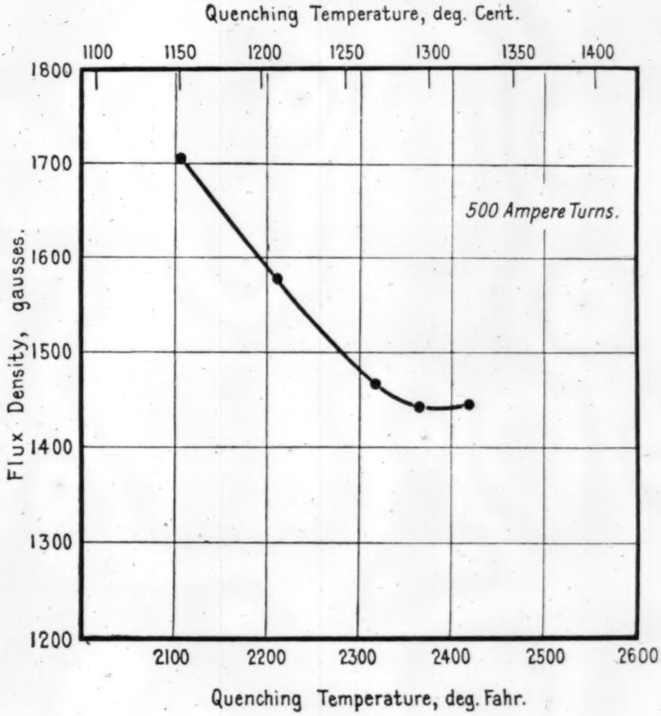

FIG. 6.—Average of Ballistic Permeability Tests with Solenoid.

A.c. Potentiometer Magnetic Test.—Tests were made with a Tinsley Gall a.c. potentiometer[1] with the specimen placed in a double solenoid 3 in. long and the secondary about 1 in. in diameter. Two sets of tests were made, one for an apparent induction in the sample of 2890 gausses and the other of 289 gausses. A diagram of connections is given in Fig. 3. The potentiometer was first connected to the secondary winding by throwing switch *S* to the left. The phase angle of the phase-shifting transformer was then changed until all of the voltage was read by potentiometer *A* (with potentiometer *B* set to zero). The balance was indicated by the vibration galvanometer *VG*. The switch was then thrown to the right connecting the potentiometer across the non-inductive resistance *R*. Potentiometer *A* then read the loss component of the current I_W and the potentiometer *B* the wattless or magnetizing component I_M.

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[1] T. Spooner, "Some Applications of the A.C. Potentiometer," *Journal*, Optical Society of America, and *Review of Scientific Instruments*, p. 217, March, 1926.

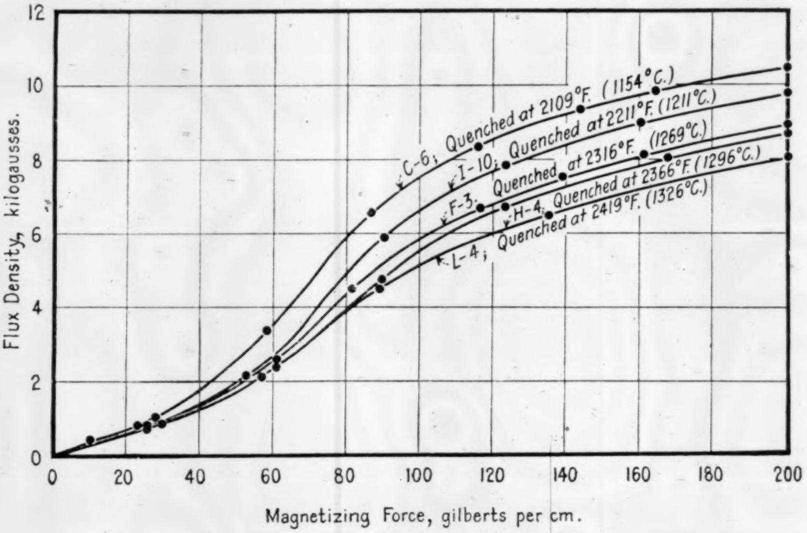

FIG. 7.—Normal Induction Curves for Representative Quenched Samples (Fahy Simplex Permeameter).

Figs. 4 and 5 show the two components plotted against quenching temperature for an apparent induction of 2890 gausses. It will be seen that the I_M values are indeterminate at the higher quenching temperatures, while the I_W values show no reversals.

Ballistic Magnetic Test.—Similar tests were made using the same coil but determining the flux densities ballistically for an applied magnetomotive force of 500 ampere turns. The results are given by Fig. 6 and are indeterminate at the higher quenching temperatures.

Fahy Simplex Magnetic Test.—In order to determine more completely the effect of induction on the relative permeability, a typical sample for each quenching temperature was selected and magnetization curves obtained by means of the Fahy simplex permeameter. The results are given by Fig. 7. It will be seen that in some cases the results are indeterminate at the lower inductions but the samples are well separated at the higher magnetomotive forces. Measurements at very low inductions corresponding to a magnetizing force of one gilbert per centimeter, however, show virtually the same order as at the high inductions.

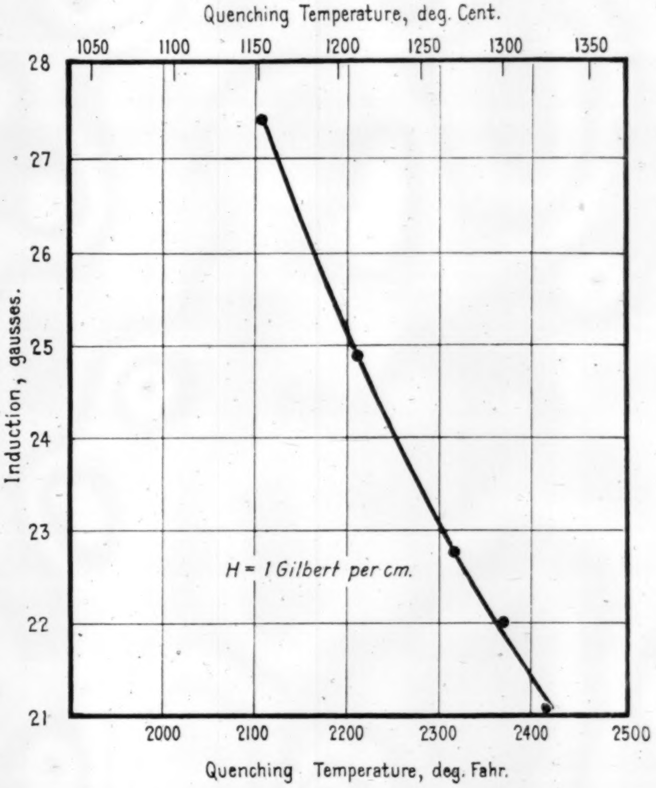

FIG. 8.—Relation of Average Induction and Quenching Temperature for Low Magnetizing Forces (Fahy Simplex Permeameter).

Fig. 8 shows such results obtained by Mr. Fahy with his d.c. simplex permeameter. The test results agree in a very satisfactory manner. Such tests are difficult to make, however, due to the small magnitude of the quantities to be measured. There is also another difficulty in connection with this low-induction test, namely, that the samples have to be very carefully demagnetized or quite erroneous results may be obtained. This demagnetization for solid material is a rather tedious process.

The necessity for demagnetization applies to a.c. as well as d.c. tests unless the inductions are rather high, provided the material has previously been subjected to any appreciable magnetic field.

Considerations of simplicity and practicability led to the use of a different type of test which could be used at higher inductions. One of the methods made use of a laminated Fahy simplex permeameter supplied with a special field winding suitable for alternating current.

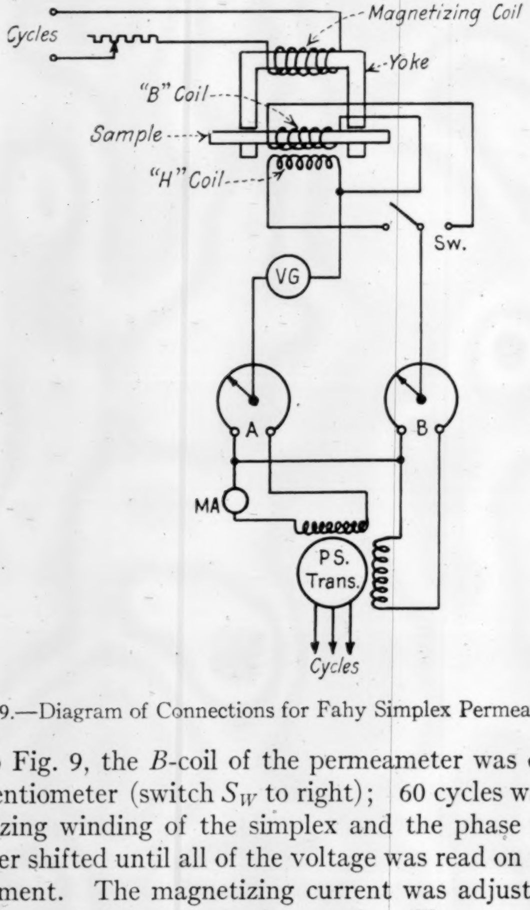

FIG. 9.—Diagram of Connections for Fahy Simplex Permeameter.

Referring to Fig. 9, the B-coil of the permeameter was connected to the a.c. potentiometer (switch S_W to right); 60 cycles was applied to the magnetizing winding of the simplex and the phase angle of the potentiometer shifted until all of the voltage was read on the A potentiometer element. The magnetizing current was adjusted to give 7 kilogausses apparent induction in the sample. The potentiometer was then switched over to the H-coil of the permeameter (switch S_W to left) and the in-phase and quadrature components of voltage read from B and A. They were called E_{HW} and E_{HM}, respectively, though they were probably not exactly proportional to the loss and magnetizing components of the a.c. field. Tan θ results were also plotted where $\tan \theta = \dfrac{E_{HW}}{E_{HM}}$. Finally the H values were plotted as calculated from the vector sum of E_{HW} and E_{HM}. Fig. 10 shows the test results.

An a.c. galvanometer instead of the potentiometer could have been used as well for a detector. It will be seen that any of these components except the E_{HW} values correlate well with the quenching temperature. The reversal of the E_{HW} values at the higher quenching temperature is probably due to several factors. In the first place as shown by d.c. data obtained by Mr. Fahy the coercive force became practically constant for quenching temperatures above 2300° F. (1260° C.) which would account for there being no further increase in E_{HW}, since hysteresis loss is proportional to coercive force. Also with the higher quenching temperature, B_r decreases. This may assist in decreasing the hysteresis loss. Finally, as the quenching temperature increases, the resistivity increases, and this would produce a

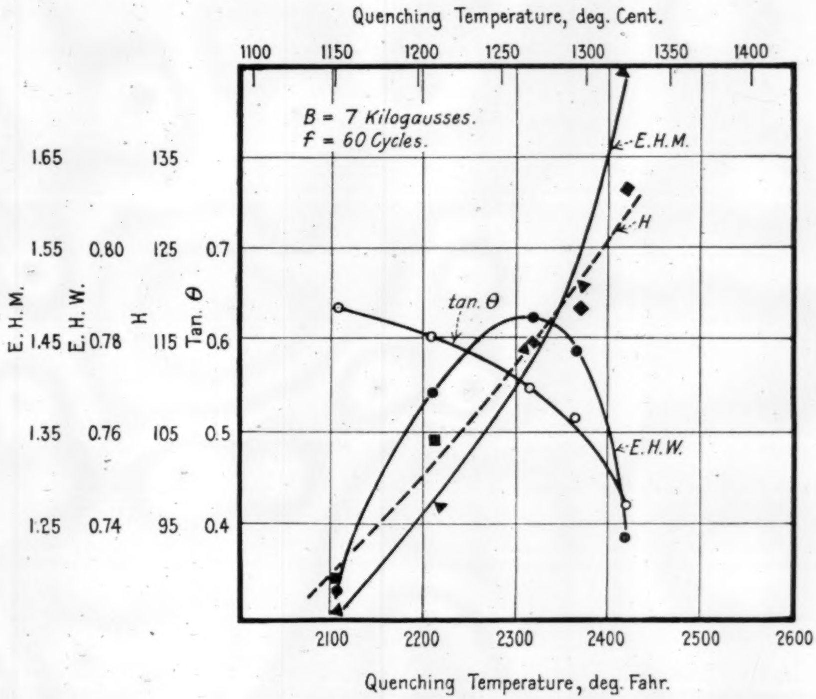

FIG. 10.—A.C. Fahy Simplex Permeameter Tests.

decrease in eddy-current loss. There are other minor factors which may also contribute to the decrease in E_{HW}.

Differential Tester.—Fig. 11 illustrates still another type of test which gave results at high inductions. This is known as a differential tester. The particular type of magnetic circuit was devised by Mr. Fahy some time ago.[1] Sixty-cycle current was applied to the center leg of a set of laminated H-shaped yokes. Across one side of the yoke was placed a standard sample having known properties and across the other the unknown sample. Two exploring coils, one on each sample, were connected differentially either to an a.c. galvanometer or an a.c. potentiometer. The field of the galvanometer was supplied from a phase-shifting transformer giving a two-phase current. The magnetizing winding on the center leg applies the same value of magnetomotive-force across the ends of both samples, assuming that the reluctance of the yokes is negligible with reference to the sample reluctances. This means that the two samples are tested under conditions of equal magnetizing force, H. What is measured then is the difference in induced voltages (see Fig. 1 (*c*)). One component is proportional to the difference in permeability of the two samples and the other to

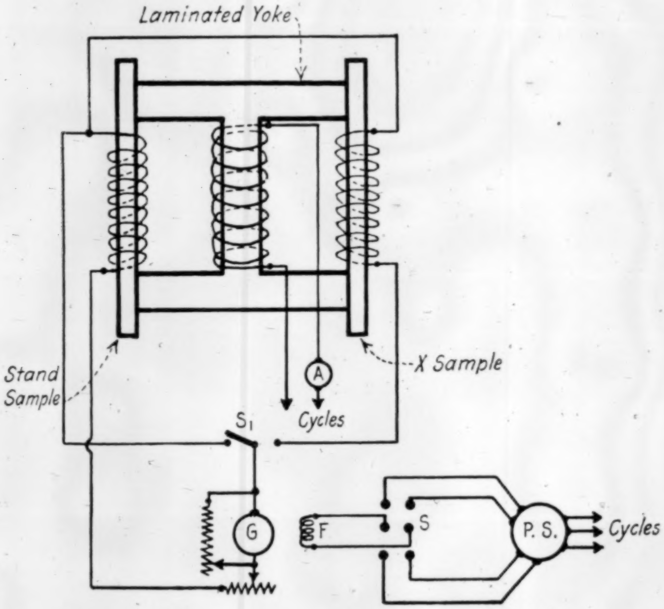

FIG. 11.—Diagram of Connections for Differential Magnetic Tester.

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[1] "An Experimental Study of the Fahy Permeameter," U. S. Bureau of Standards *Scientific Paper No. 360*, 1917.

the difference in losses. The loss components are also a function of the permeability since the tests are made for equal H values and therefore the inductions in the standard and test samples are different.

The method of test was as follows: The exploring coil on the standard sample was connected to the galvanometer (switch S_1 left) and the phase-shifting transformer rotated until with switch S up, for instance, the galvanometer read zero. With S down it would then read a maximum. The two exploring coils were next connected differentially to the galvanometer (switch S_1 right) and the deflections

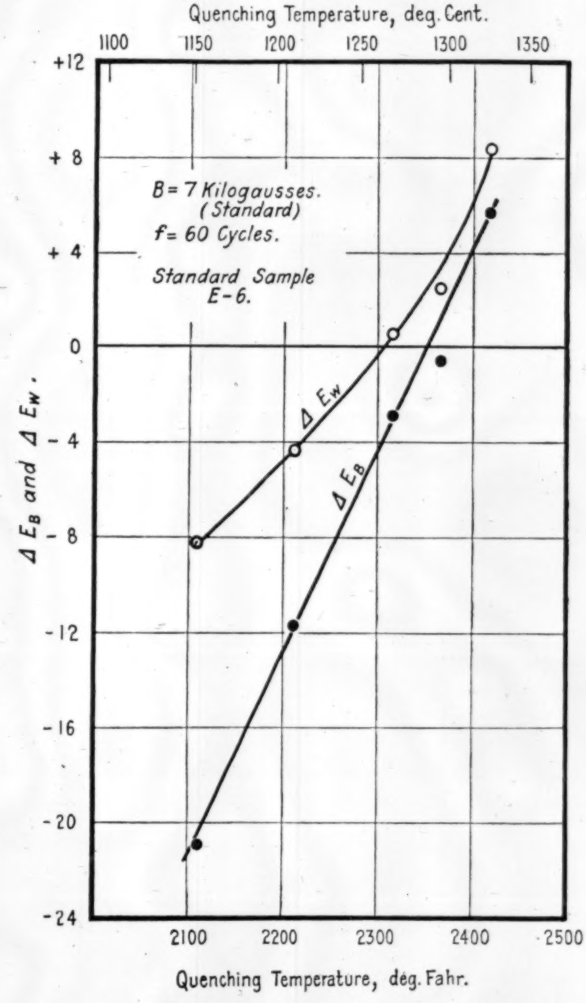


FIG. 12.—Average Results of Differential Tests with A.C. Galvanometer.

noted with S in both positions. One reading gave the voltage proportional to the difference in loss of the two samples (ΔE_W) and the other proportional to the difference in induction (ΔE_B).

Fig. 12 shows the average results plotted against quenching temperature for the differential tester.

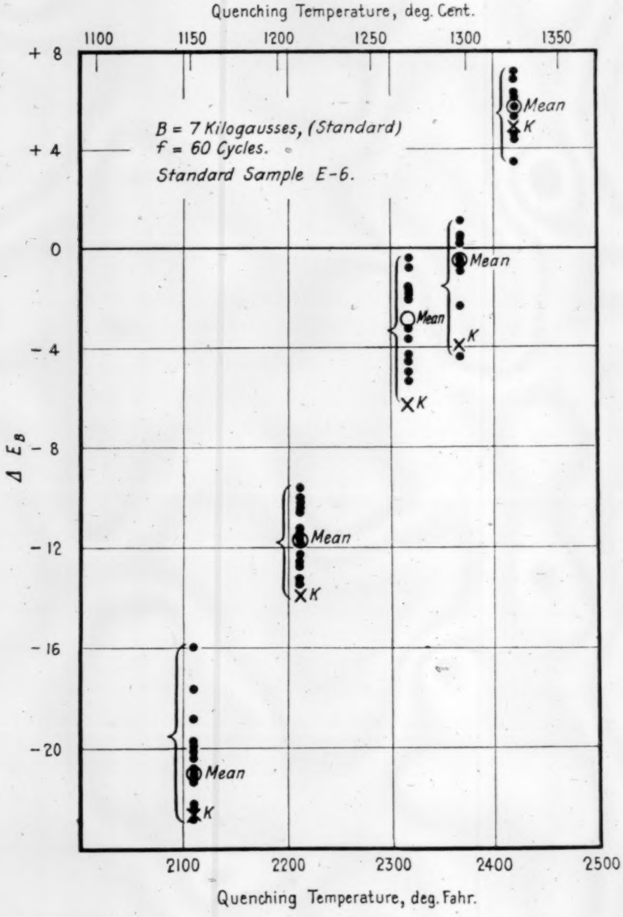


FIG. 13.—Induction Components of Individual Samples in Differential Tests with A.C. Galvanometer After Quenching.

Figs. 13 and 14 show the individual points. It will be noted that in the case of the E_W values there is no definite overlap for the different temperatures with the exception of one 2366° F. (1296° C.) sample which was probably quenched at too low a temperature; and of course the K samples which were quite different chemically.

This differential tester gives a very quick and simple means of determining the uniformity of quenching temperature. If the magnetizing coil is connected to a steady supply of voltage such as a commercial lighting circuit no adjustment of current would ordinarily need to be made, since the readings are independent of ordinary changes in voltage.

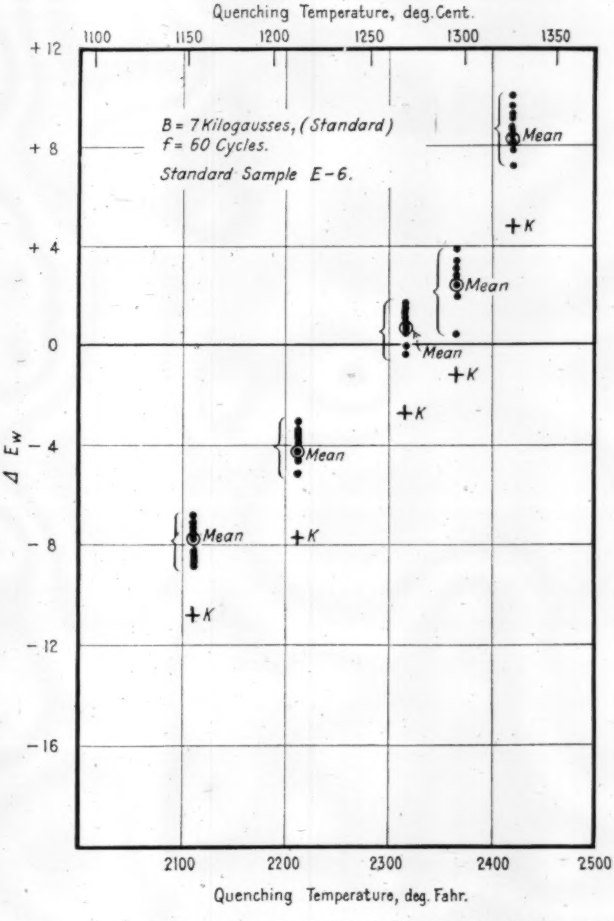
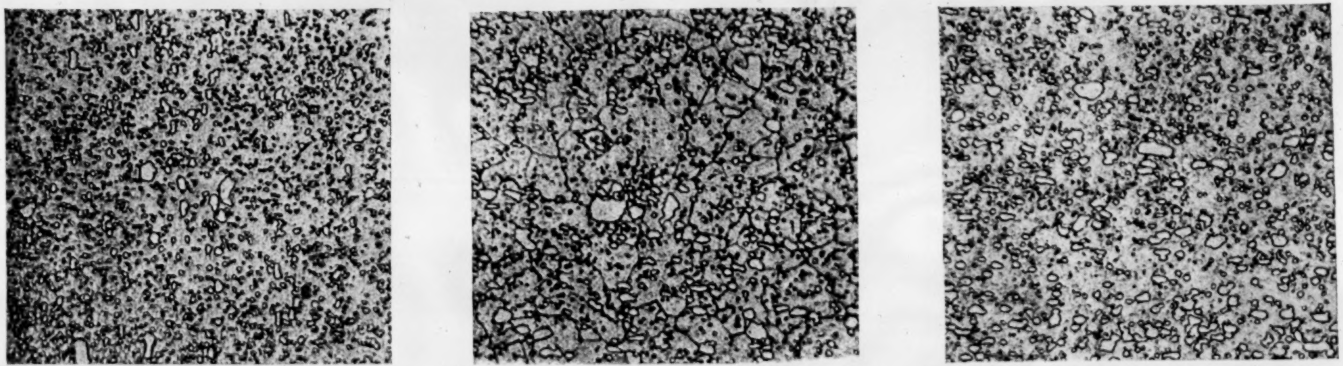


FIG. 14.—Loss Components of Individual Samples in Differential Tests with A.C. Galvanometer After Quenching.

This same apparatus using the a.c. potentiometer as an indicator gave similar results.

Specimen K-10, Normalized.

Specimen G-4, Normalized.

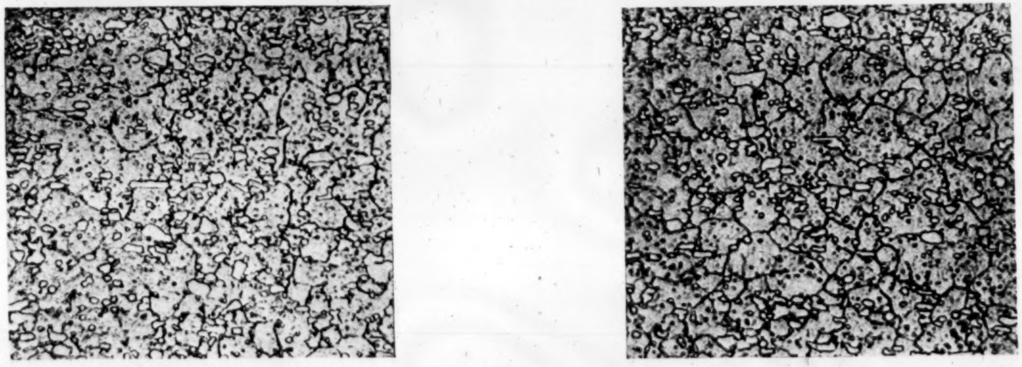


Specimen K-8, Quenched from 2109° F. (1154° C.)

Specimen B-2, Quenched from 2109° F. (1154° C.).

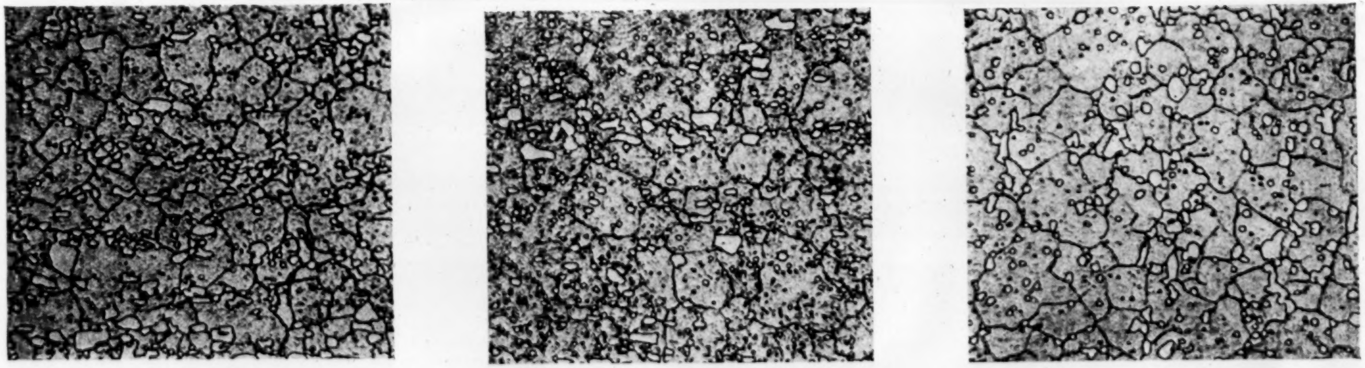
Specimen C-6, Quenched from 2109° F. (1154° C.).

FIG. 15.—Photomicrographs of Normalized and Quenched High-Speed Steel. (× 500). Etching solution, 1 per cent nitric acid in alcohol and then Kourbatoff's reagent consisting of 4 parts hydrochloric acid, 20 parts iso-amyl alcohol and 75 parts alcoholic solution of nitroaniline.



Specimen C-1, Quenched from 2211° F. (1211° C.).

Specimen E-2, Quenched from 2211° F. (1211° C.).

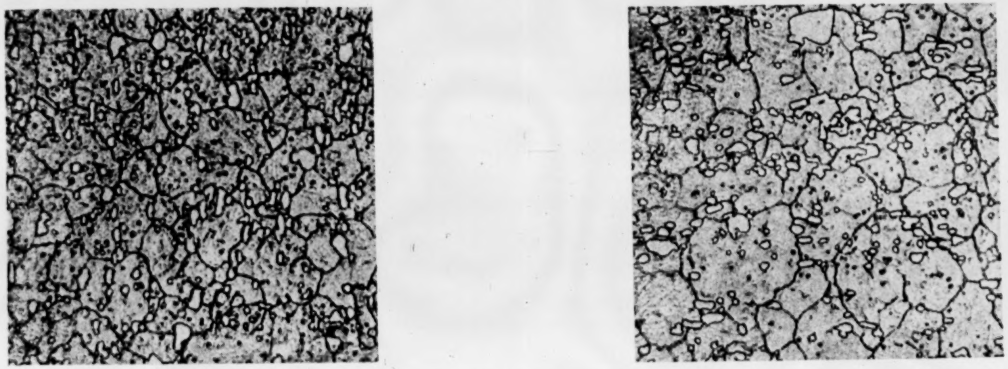


Specimen B-10, Quenched from 2316° F. (1269° C.).

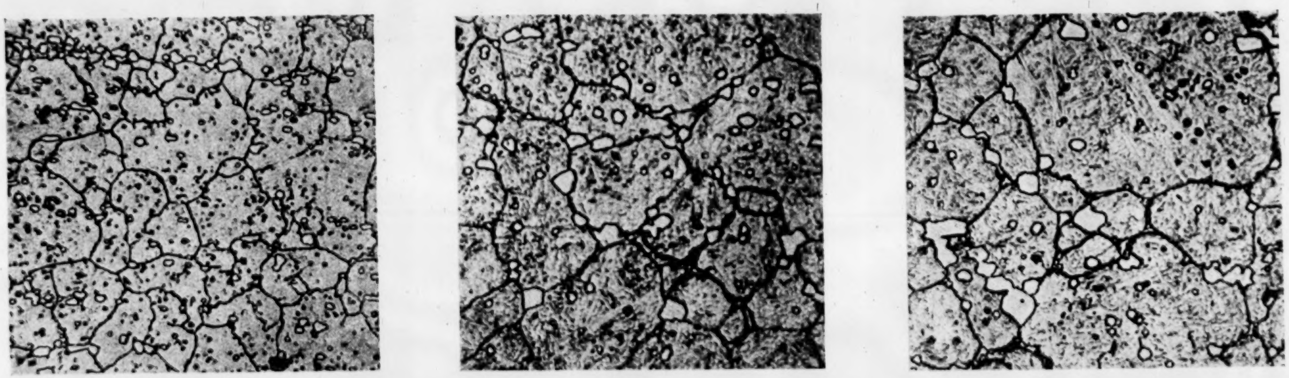
Specimen D-7, Quenched from 2316° F. (1269° C.).

Specimen A-6, Quenched from 2316° F. (1269° C.).

FIG. 16.—Photomicrographs of Quenched High-Speed Steel. (× 500). Etching solution, 1 per cent nitric acid in alcohol and then in Kourbatoff's reagent, consisting of 4 parts hydrochloric acid, 20 parts iso-amyl alcohol, 75 parts alcoholic solution of nitroaniline.

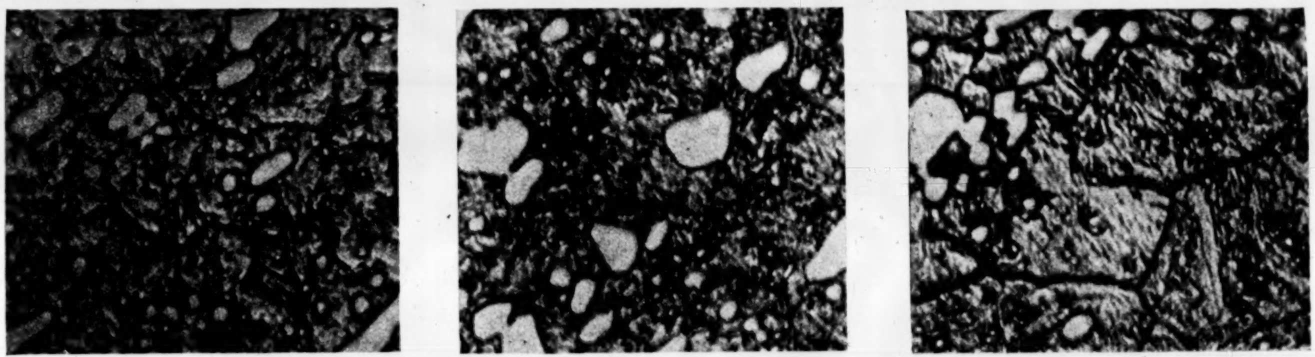


Specimen D-6, Quenched from 2366° F. (1296° C.). Specimen E-7, Quenched from 2366° F. (1296° C.).



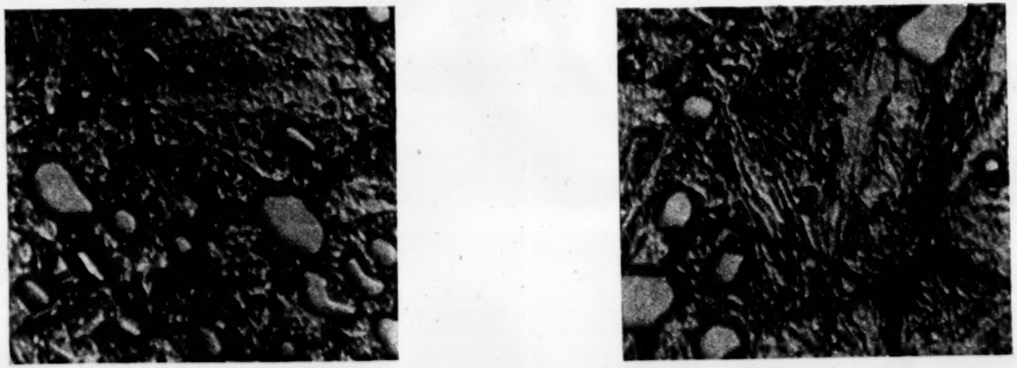
Specimen K-1, Quenched from 2366° F. (1296° C.). Specimen H-5, Quenched from 2419° F. (1326° C.). Specimen I-5, Quenched from 2419° F. (1326° C.).

FIG. 17.—Photomicrographs of Quenched High-Speed Steel. (× 500). Etching solution, 1 per cent nitric acid in alcohol and then in Kourbatoff's reagent, consisting of 4 parts hydrochloric acid, 20 parts iso-amyl alcohol, 75 parts alcoholic solution of nitroaniline.



Specimen B-2, Quenched from 2109° F. (1154° C.). Specimen E-2, Quenched from 2211° F. (1211° C.). Specimen B-10, Quenched from 2316° F. (1269° C.).

FIG. 18.—Photomicrographs of Quenched High-Speed Steel. (X 2000). Etching solution, Kourbatoff's reagent, consisting of 4 parts hydrochloric acid, 20 parts iso-amyl alcohol, 75 parts alcoholic solution of nitroaniline.



Specimen E-7, Quenched from 2366° F. (1296° C.). Specimen I-5, Quenched from 2419° F. (1326° C.).

FIG. 19.—Photomicrographs of Quenched High-Speed Steel (X 2000). Etching solution, Kourbatoff's reagent, consisting of 4 parts hydrochloric acid, 20 parts iso-amyl alcohol, 75 parts alcoholic solution of nitroaniline.

Photomicrographs on Quenched Material:

Certain of the quenched bars were examined microscopically under the direction of Mr. H. Styri of the SKF Laboratories. The work was done by Mr. Walp. Certain typical photomicrographs at magnifications of 500 are reproduced in Figs. 15, 16 and 17.

A comparison of the K (14 per cent tungsten) with the normal specimens (17.3 per cent tungsten) is interesting. For the same actual quenching temperatures, the K samples appear to have been quenched at a lower temperature than the normal samples. The magnetic results show the same differences.

A comparison of samples B-2 and C-6 is instructive. Specimen B-2 shows definite grain boundaries whereas C-6 does not. This would indicate that B-2 was quenched at a higher temperature. This is undoubtedly the case since the magnetic results show the same thing, namely, B-2 is the hardest magnetically of all of the samples (with one exception) which were quenched at 2109° F. (1154° C.).

The difference between the 2211, 2314 and 2366° F. (1211, 1268 and 1296° C.) quenches are very difficult to detect with certainty from the photomicrographs, but the differences magnetically are very pronounced. The large increase in grain size and the thicker grain boundaries for the 2419° F. (1326° C.) quench should be noted.

Figs. 18 and 19 show typical photomicrographs for a magnification of 2000. They were obtained also by the SKF Laboratories. The structure of the individual grains is brought out much clearer by the higher magnification.

Hardness Tests on Quenched Material:

Rockwell C hardness tests were made on some of the quenched samples by Mr. Walp of the SKF Laboratories with the following average results:

TABLE I.—HARDNESS TESTS.

QUENCHING TEMPERATURE DEG. FAHR.	DEG. CENT.	ROCKWELL C HARDNESS
Normalized		22.6 (K sample 10.7)
2109	1154	64.9 (K sample 60.1)
2211	1211	65.9
2316	1269	66.1
2366	1296	65.8 (K sample 64.9)
2419	1326	65.2

It will be seen that the Rockwell hardness test is a very poor indication of the quenching temperature over the range from 2100 to 2400° F. (1149 to 1316° C.).

OTHER QUENCHING TESTS

Methods and Results.—In order to determine the effect of time in the high-speed bath, another series of bars was quenched as before from 2350° F. (1288° C.), but varying the time at the quenching temperature from 1 to 30 minutes. In general, three bars were quenched for each time, except for the longest intervals, in order to eliminate accidental variations. Fig. 20 shows the results as obtained on the differential tester using the a.c. potentiometer as an indicator. The standard sample was held at 7 kilogausses, 60 cycles. By noting that the voltage induced in the standard coil was 0.726, the magnitude of the variations may be estimated. The quenching temperatures varied somewhat from the normal value of 2350° F. (1288° C.). ΔE values were corrected to the standard temperature by means of curves previously obtained. These corrections may not have been altogether accurate which would account, in part, for the fact that the points for a given time vary somewhat.

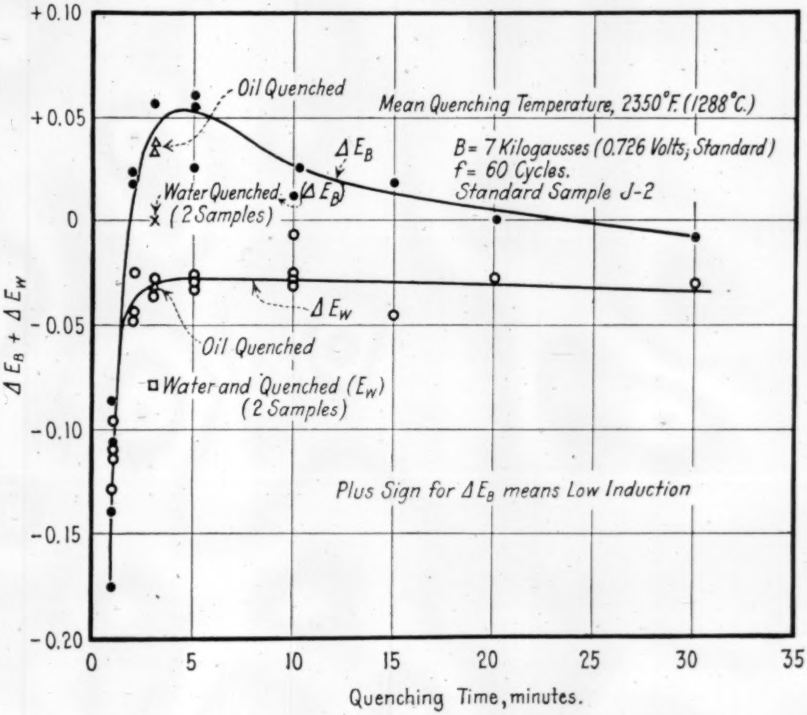


FIG. 20.—Induction and Loss Components in Differential Tests with A.C. Potentiometer. Effect of Time in High-Speed Bath.

In addition to the bars which received the normal treatment, two samples were quenched in oil directly, eliminating the intermediate quench in the salt bath; also two samples were quenched in water directly from the high-speed bath.

Discussion of Results Obtained with Varying Time of Quench.—The curves in Fig. 20 are very significant. In analyzing them it should be noted that a plus sign for ΔE_B means a decreased induction or permeability with reference to the standard. Obviously, a certain minimum time is required to bring the specimen up to the temperature of the high-speed bath. If the time is too short, the sample will be quenched at such a low temperature that the carbon cannot all be absorbed, with the result that less than a normal amount of retained austenite would be formed. This would be indicated by a minus value of ΔE_B. Also, even though the sample came to temperature, an appreciable time is required for the absorption of carbon and for other changes. It is evident from the curves that one minute is far from long enough in the quenching bath; two minutes is a little too short. The three minutes which was normally used gave very good results although perhaps four minutes would have been as well or better.

For other samples, the necessary time might be quite different. For a larger cross-section or larger ratio of mass to surface area the time should, of course, be greater.

It will be noted that there is a decrease in the ΔE_B values for the longer times in the high-speed bath. This is due to the surface decarbonization which begins to become appreciable after 5 minutes.

It may be noted that the direct oil-quenched samples gave practically identical results with those samples having the double quench, but the water-quenched samples showed a higher induction, indicating a greater percentage of martensite. This is in line with Mathews'[1] recent theories.

The advantages of the double quench are that possibly it has less tendency to produce cracking and the low-temperature salt is much easier to remove from the steel than the high-speed salt.

Such a magnetic test as this provides a very quick method of obtaining data as to the most suitable time of heating at the quenching temperature.

EFFECTS OF DRAWING

Drawing Procedure.—The 75 bars which were quenched at 2109, 2211, 2314, 2366 and 2419° F. (1154, 1211, 1268, 1296 and 1326° C.) were drawn in a Bellis salt bath at 1000, 1050, 1100 or 1150° F. (538, 566, 593 or 621° C.), except that three bars from each quenching temperature were left undrawn. In the final state there were three bars for each pair of drawing and quenching temperatures. The time in the drawing bath was 30 mintues, 6 bars being drawn simultaneously. The temperature was controlled manually as indicated by a calibrated base-metal thermocouple. The mean temperatures are believed to have been correct to ±3° F. (1.5° C.), with a maximum variation of ±8° F. (5° C.), the average variation being less than half of this.

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[1] J. A. Mathews, "Retained Austenite," *Transactions*, Am. Soc. Steel Treating, p. 565, November, 1925.

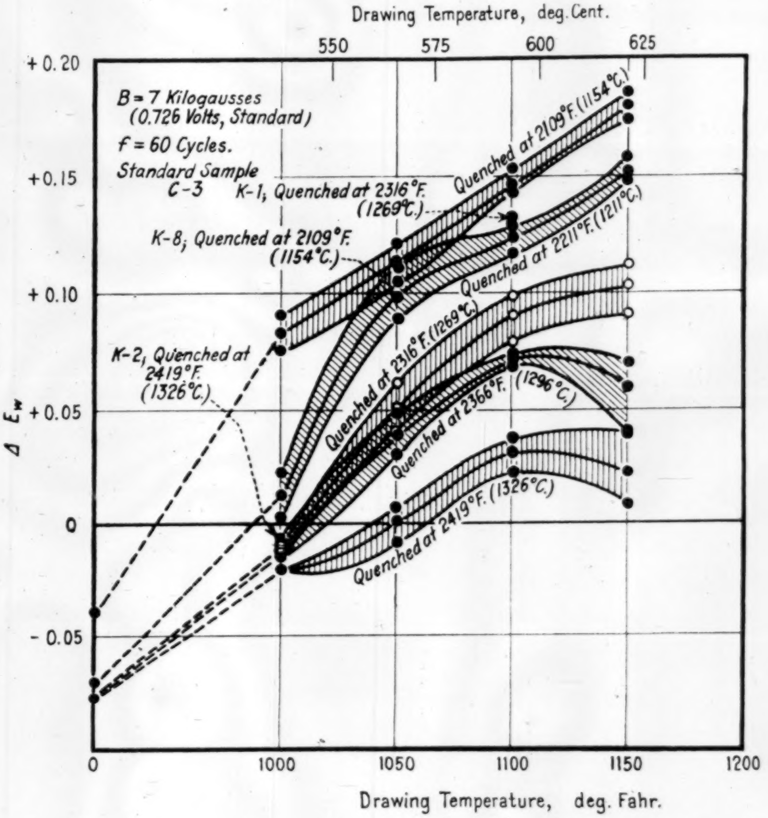

FIG. 21.—Induction Components, After Drawing, in Differential Tester with A.C. Potentiometer.

Tests after Drawing.—After drawing, the bars were tested in the differential tester at 60 cycles, using the a.c. potentiometer as an indicator. In general, not more than 30 minutes elapsed before testing after the bars were removed from the drawing bath. The bars were retested at intervals to determine the aging. This will be discussed later.

After some days, when the bars had settled down to a steady state, they were tested at 60 cycles in the a.c. Fahy simplex permeameter, using the a.c. potentiometer as an indicator. Two apparent inductions were used, namely, 7 kilogausses and 10 gausses. The two components of the voltage of the H coil, E_{HW} and E_{HM} were measured as previously described.

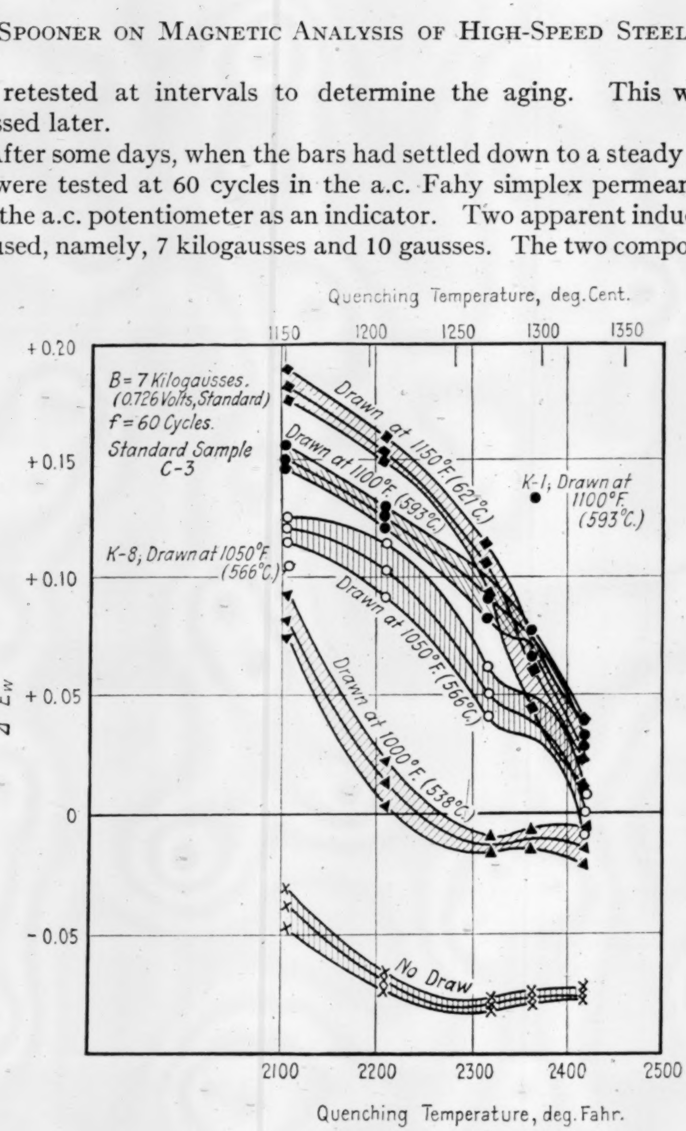

Fig. 22.—Induction Components, After Drawing, in Differential Tester with A.C. Potentiometer.

Fig. 21 gives the difference in one component of secondary voltage (ΔE_W) between the standard and test samples plotted against drawing temperature, using the differential tester. ΔE_W is the component of voltage in quadrature with the voltage induced by the standard sample

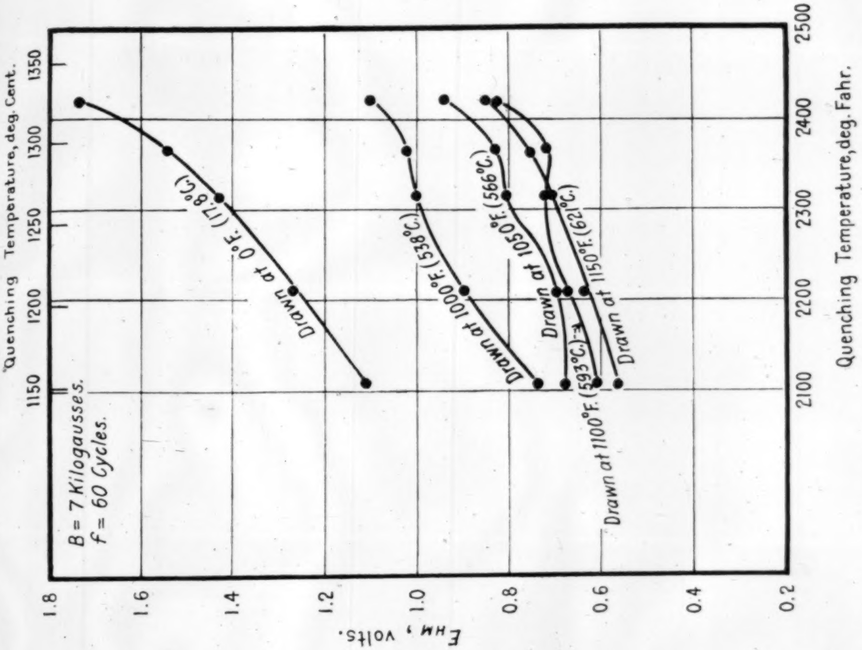

FIG. 24.—Magnetizing Component, After Drawing, in A.C. Simplex Permeameter with A.C. Potentiometer.

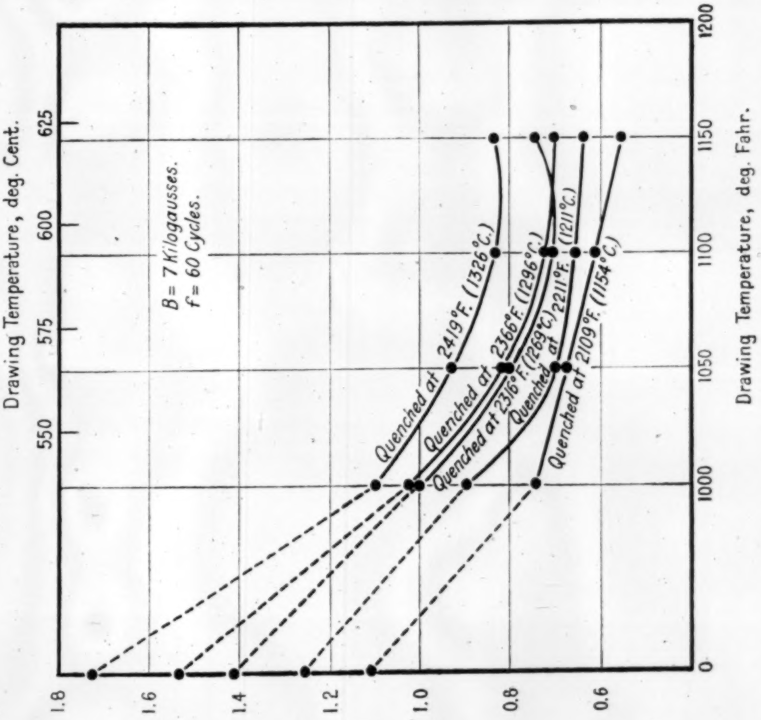

FIG. 23.—Magnetizing Components, After Drawing, in A.C. Simplex Permeameter with A.C. Potentiometer.

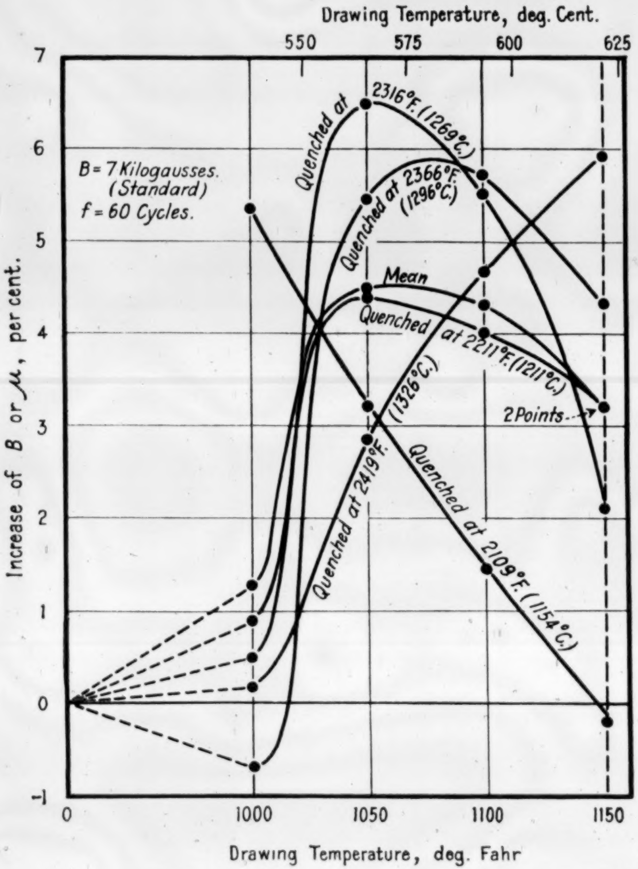

FIG. 25.—Showing the Effect of Aging After Drawing at the End of 4 Days.

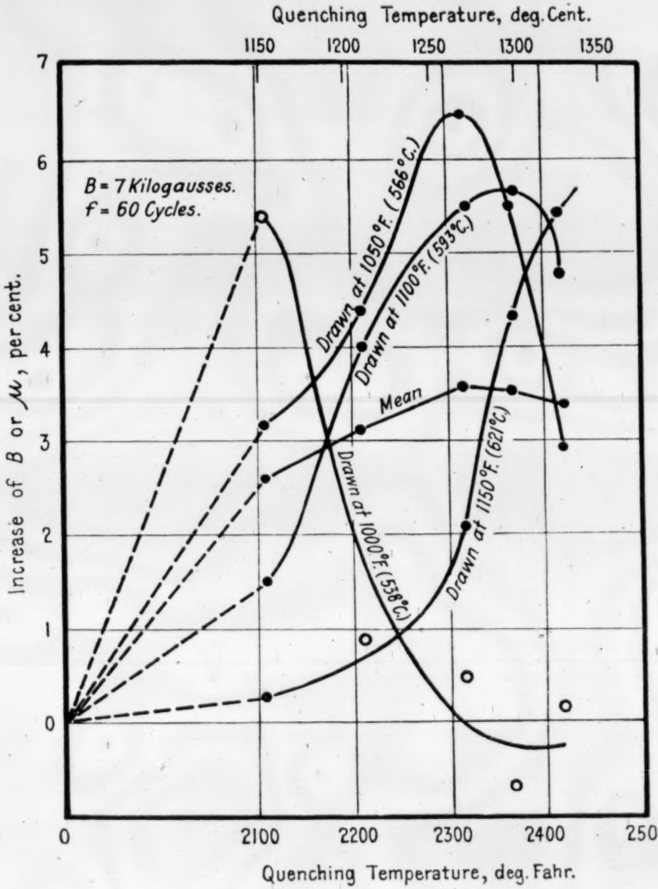

FIG. 26.—Showing the Effect of Aging After Drawing at the End of 4 Days.

for an apparent maximum induction of 7 kilogausses in the standard sample. If comparisons of absolute magnitude are to be made, it should be noted that 0.726 volts was induced by the standard bar. The cross-hatched areas indicate the spread of the three samples of each set having the same heat treatment. The center line corresponds to the mean values.

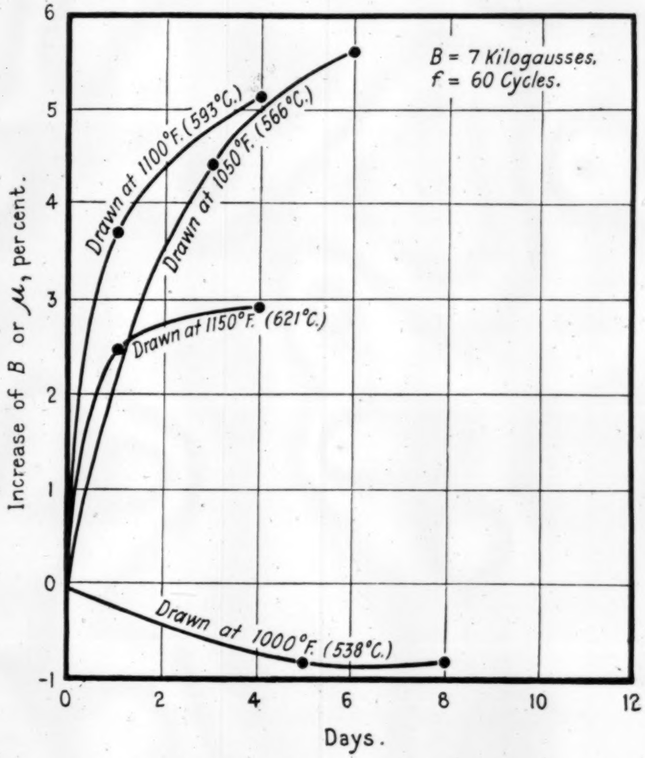

FIG. 27.—Showing Relation Between Aging and Time for Various Drawing Temperatures. Quenching Temperature, 2366° F. (1297° C.).

Fig. 22 gives the same results as given by Fig. 21 plotted against quenching temperature.

Fig. 23 gives the simplex permeameter magnetizing components of H (E_{HM}) plotted against drawing temperature for an apparent maximum induction of 7 kilogausses and Fig. 24 gives the same data plotted gainst quenching temperature. Similar results were obtained for an apparent induction of 10 gausses.

Aging after Drawing.—Fig. 25 shows the average aging of the samples after the drawing operation in percentage increase of B or μ

for an apparent induction of 7 kilogausses plotted against drawing temperature. The aging is for a period of 4 days. Fig. 26 shows the the same results plotted against quenching temperature.

Fig. 27 shows some typical aging data plotted against time for a 2366° F. (1296° C.) quenching, and Fig. 28 shows similar data for a 1050° F. (566° C.) draw. These data will not quite check the results of Figs. 25 and 26 since they did not include all of the samples.

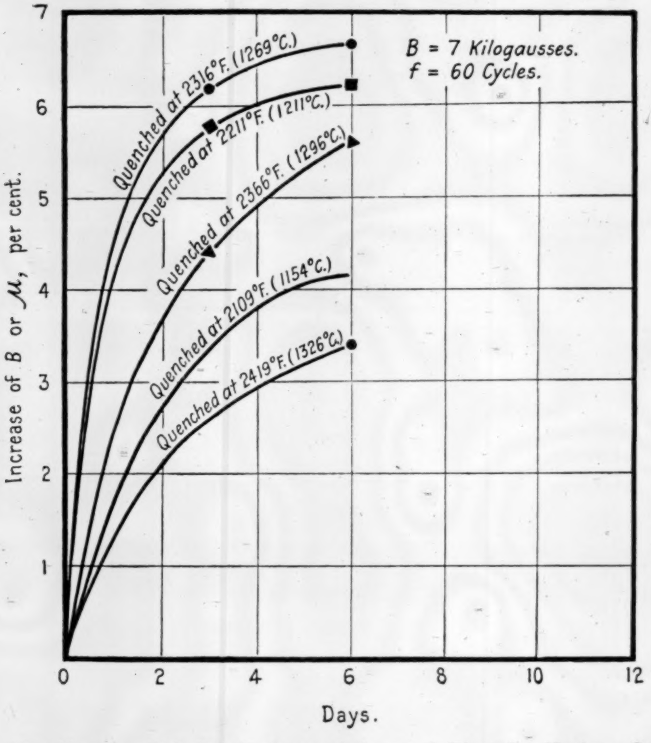

FIG. 28.—Showing Relation Between Aging and Time for Various Quenching Temperatures. Drawing Temperature, 1050° F. (566° C.).

It may be noted that the aging results for individual samples varied rather widely. Each point on the curve for Figs. 25 and 26 is the average for three bars and therefore the shape of the curves should be fairly representative. Tests made on some of the bars at the end of from 15 to 19 days indicated that the change subsequent to 4 days was rather small and probably little greater than the accuracy of testing. It may be noted finally that, except for the 1000° F. (538° C.) draw, the change is in the direction of a softening, due probably either to a further precipitation of carbides or to a removal of strains, or both.

Examination for aging after quenching gave results somewhat similar to those obtained after drawing. The values are not directly comparable, however, since a different method of test was used.

Discussion of Results after Drawing.—Figs. 21 and 22 show that for a given quenching temperature a magnetic test will indicate the drawing temperature in a fairly reliable manner, except that for a 2366 and a 2419° F. (1296 and 1326° C.) quench there is little difference between a 1100 and a 1150° F. (593 and 621° C.) draw. In fact, between these two temperatures there is a slight reversal of the magnetic properties, probably corresponding to the secondary hardening.

For a given drawing temperature ΔE_W (Fig. 22) gives in general a very sensitive indication of the previous quenching temperature. This is especially true for the higher drawing temperatures.

The spread between the various samples is not very large, indicating that the heat treatment was fairly uniform. The three samples corresponding to each quench were drawn at different times.

The differences between the various samples after the final heat treatment are probably little, if any, greater than for the original normalized bars. This cannot be determined definitely for the high-induction test since this particular type of test was not made on the normalized bars.

The tests on the simplex permeameter showed a spread for the individual bars of the same order of magnitude as for the differential tester. Only the mean values were plotted for this test, however. Here again the results are rather indeterminate between the higher drawing temperatures for the higher quenching temperatures.

The tests with the solenoid at moderate inductions (2890 gausses are not so satisfactory as the other tests for distinguishing between the various heat-treating temperatures, since the solenoid curves have a greater tendency to cross each other and show greater peculiarities. It is probable that for this range of inductions, the effects of stress complicate the results.

The aging effects have been discussed previously. Evidently, changes of considerable magnitude take place at room temperatures after drawing. The magnetic test makes a very simple method of following these changes. A further study will be necessary in order to interpret their significance. Somewhat similar effects occurred after quenching and in the same direction, but the results cannot be compared directly with the aging after drawing since a different method of test was used in the two cases.

CONCLUSIONS

As shown previously, high-induction magnetic tests—except perhaps in the case of annealed material—seem to be preferable to tests at moderate inductions. This is probably due to the fact that at moderate inductions the magnetic properties are a function not only of the chemical composition and metallographic constitution but are largely affected by physical strains. At high inductions the effect on the magnetic properties of physical strains become much less or sometimes negligible. The disturbing effect of physical strains is probably responsible, to a considerable degree, for the partial failure of certain previous investigations made for the purpose of correlating magnetic properties with heat treatment. In some cases it is desirable to have a measure of the strains, but when it is preferable to eliminate their effect a high-induction test furnishes the means.

The above results indicate then that, except for a certain limited combination of quench and draw, a fairly high-induction, 60-cycle test, gives a sensitive indication of the uniformity of the drawing treatment for high-speed steel bars of the size here considered. If the quenching temperature is known, the actual drawing temperature can be determined magnetically quite accurately (see Fig. 21).

If the drawing temperature is known, the preveious quenching temperature can be determined fairly closely for drawing temperatures of 1050° F. (566° C.) or above (see Fig. 22).

The magnetic tests are quick and simple, and the results are quite reproducible. The magnetic tests make a simple and sensitive method for following changes in the material after heat treatment. These changes after quenching and drawing continue for some days and are apparently of considerable magnitude, especially after the drawing operation.

These results warrant attention being directed to the greater commercial use of magnetic tests as a means of determining:

1. The uniformity of material before heat treatment, both chemically and as a function of mill practice;
2. The uniformity of the quenching operations to check not only temperature but oxidation and decarbonization; and
3. The uniformity of drawing operations.

In many cases a single test on the completely heat-treated specimens will indicate the cause of variations from the normal. It is safer and probably more satisfactory, however, for the manufacturer of tools to check the material after each operation in order to determine definitely the cause of any variations which may appear, and correct the fault as soon as possible.

Finally, the desirability is urged of applying a magnetic test to a series of heat-treated physical test specimens when such specimens are to be used for endurance tests, either at normal or elevated temperatures. A simple magnetic test may save months of wasted time due to lack of uniformity of the samples.

Certain desirable data are missing from this paper, such as photomicrographs after drawing and tests at very high inductions showing the reluctivity relationship. It is hoped that these deficiencies will be supplied by other investigators who have obtained such data on this high-speed material. It would be very desirable for the magnetic results to be correlated with actual machining tests. It is possible that at a later date this can be done.

[For a Discussion on Magnetic Analysis, see page 155.—ED.]

TESTING OF BALL BEARING RACES BY ELECTRIC AND MAGNETIC METHODS

By Haakon Styri[1]

Synopsis

Both electric and magnetic methods can be used in testing steel for control of heat treatment. The electric resistance can often be easily measured. For ball bearing rings, magnetic methods seem more practical. Of many possible arrangements, one using the ring as magnet core, the other using the ring as short-circuited secondary in a transformer, are of particular interest. The latter gives the quicker method for comparative testing, and with properly selected standard and proper phase relation between field of galvanometer and core, very good differentiation between hardened rings can be obtained. Two or more phase readings will approximately identify the heat treatment.

In the testing of ball bearing rings it is clearly desirable, first, that they be tested directly after heat treating, without necessitating any grinding operation; second, that the testing method and apparatus be suitable for a large variety of sizes; third, that the testing method be sufficiently rapid, and of course, that it be sufficiently accurate.

A rotating hysteresis apparatus on similar principles as described by Sanford and Fischer,[2] was tried, but it was found difficult to make an apparatus rugged and sensitive enough for the shop and suitable for different sizes of rings. Better results were obtained by a simple measurement of the electric resistance of the rings, which, as is well known, is a function of the heat treatment. A current of known value can be sent through a ring between two diametrically opposite contact points, and the potential drop between two points measured with a millivoltmeter (Fig. 1). Sharp steel points under pressure give sufficiently good contact for the voltmeter, but it is not necessary that the current contacts be particularly prepared, so a ring can be tested directly after quenching. When arrangements are made to maintain a constant current, it is necessary to make only one reading for the determination of the resistance, which in turn indicates the heat treatment, as shown in Fig. 4. This method undoubtedly has value, but on account of some objectionable features it has not been further developed.

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[1] Chief of Research Laboratory, S. K. F. Industries, Inc., Philadelphia, Pa.

[2] R. L. Sanford and M. F. Fischer, "Application of Magnetic Analysis to the Testing of Ball Bearing Races," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, p. 68 (1919).

It seemed more promising to use the rings in some kind of a transformer arrangement. Of the many possible forms, two differing in principle have already been referred to by Heindlhofer in an earlier paper.[1] One of these arrangements has the ring as magnet core, the other has the ring as a secondary winding. For the first of the arrangements a standard wattmeter was originally used to measure the secondary current, while the voltage terminals of the wattmeter were connected to a constant voltage from one or another phase in the two-phase supply (Fig. 2). One switch closed both primary and secondary windings. Differences in heat treatment could be shown by this arrangement, but as the amplitude of the secondary voltage would be nearly alike for hardened and annealed rings, although the phase would be different, due to the simultaneous change of resistance and permeability with heat treatment, it was necessary to use certain phases in the voltage terminals of the wattmeter as reference in order to identify differently heat-treated rings. The results of tests with this method are also shown in Fig. 4. The wattmeter available consumed considerable energy and heavy currents had to be used to get readings. The rings would therefore tend to heat up and affect the readings, and only fairly large rings could be tested.

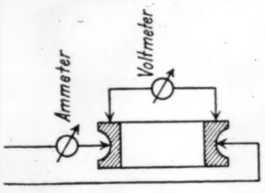

FIG. 1.—Measurement of Electrical Resistance of Ring.

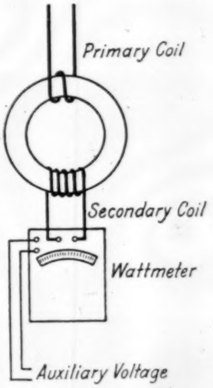

FIG. 2.—A. C. Method Using Circular Magnetic Flux.

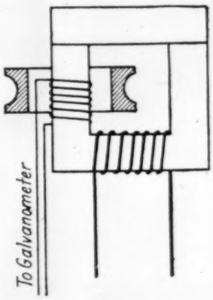

FIG. 3.—A. C. Method Using Circular Induced Current.

The other method, with the ring as short-circuited secondary (Fig. 3) seemed preferable, and although only a fairly sensitive thermo-galvanometer (Weston 425) was available to begin with, it was possible easily to distinguish annealed and hardened rings by one reading.

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[1] *Iron Age*, 1925, p. 606.

A constant current of 5 amperes was sent through the primary winding, giving about 40 ampere per sq. cm. cross-section of the ring, and the Weston galvanometer was connected to a secondary search coil of 8 windings. For any given set of rings there would be an optimum number of primary ampere turns, which would give the greatest difference in galvanometer deflection, because the impedance of hard rings would increase slowly and uniformly with increasing ampere-turns, while the impedance of soft rings would rise rapidly to a maximum and then decrease.

The following data were obtained with this method. Rings about 70 mm. in average diameter with cross-section about 4 sq. cm. quenched in water from 1454° F. (790° C.) and drawn at various temperatures gave the following scale readings:

DRAWN AT	SCALE READINGS
212° F. (100° C.)	29
320° F. (160° C.)	32
419° F. (215° C.)	43
Annealed	125

In comparison, a thin ring quenched in oil from 1550° F. (843° C.) split open and ground for careful measurements gave the following resistances as quenched:

DRAWN AT	RESISTANCE
	34.5 microhm per cm^3
212° F. (100° C.)	33.2 " " "
300° F. (149° C.)	31.4 " " "
351° F. (177° C.)	29.8 " " "
Annealed	21. " " "

However, sufficient differentiation between hardened rings could not be obtained, and, also, to take readings would require too long a time for shop use, because a closed yoke was necessary with this instrument.

A much more sensitive instrument would be necessary for a more convenient method which would permit measuring smaller variations by means of a differential method. The author learned that Leeds & Northrup had built one for de Forest, who very kindly showed him the arrangement for magnetic testing, later described before this Society in 1923.[1] De Forest also made some measurements with the ring as magnet core and could easily show magnetic variation around the circumference of the ring, using only one primary and one secondary winding. Differences in structure could, in many cases, be verified by microscopic examination.

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[1] A. V. de Forest, "A New Method of Magnetic Inspection," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 611 (1923).

This method of testing was very interesting and useful, but somewhat slow. It seemed also more desirable to measure the average properties, so it was decided to select for further development the second transformer arrangement mentioned above, having the ring as a short-circuited secondary.

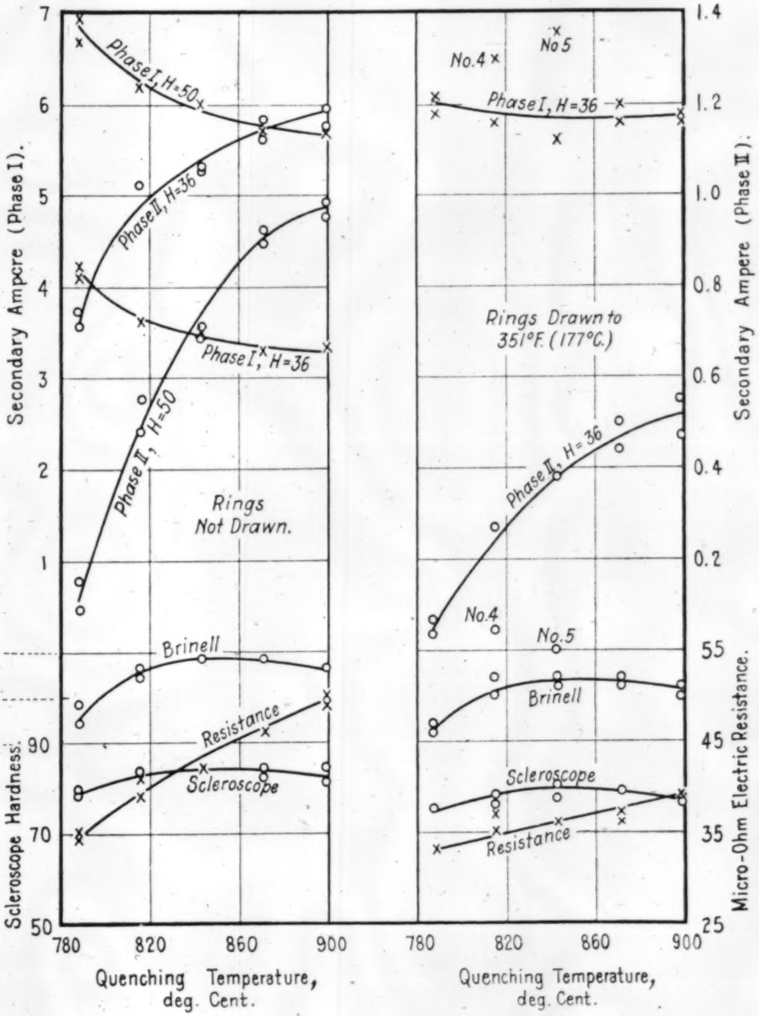

FIG. 4.—Results of Tests Using Methods Shown in Figs. 1 and 2.

The field of the Leeds & Northrup No. 2470 galvanometer could be connected to one or another phase of the two-phase supply, which

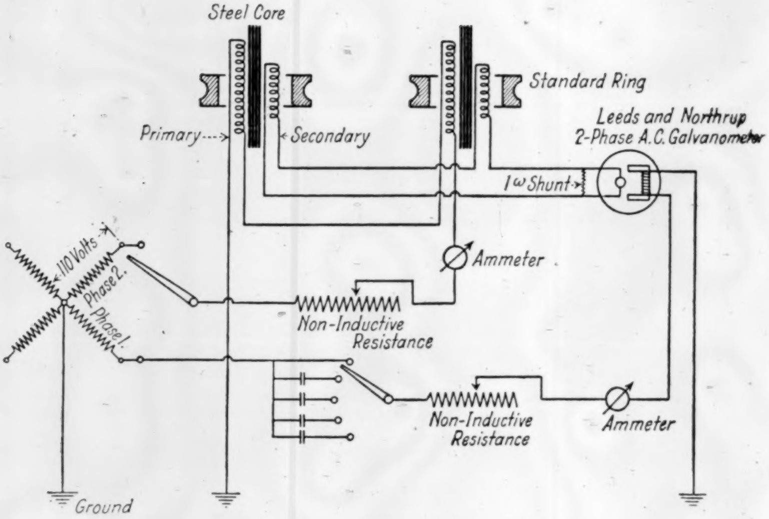
Steel Core
Standard Ring
Primary
Secondary
Leeds and Northrup
2-Phase A.C. Galvanometer
1 ω Shunt
110 Volts
Phase 2.
Phase 1.
Ammeter
Non-Inductive
Resistance
Non-Inductive
Resistance
Ammeter
Ground

FIG. 5.—Wiring Diagram of Magnetic Tester for Ball Bearing Races.

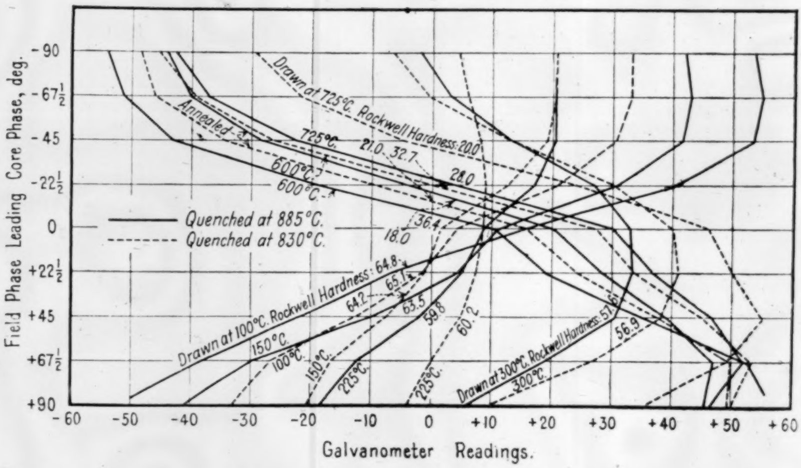

FIG. 6.—Results of Tests at Various Phase Relationships.

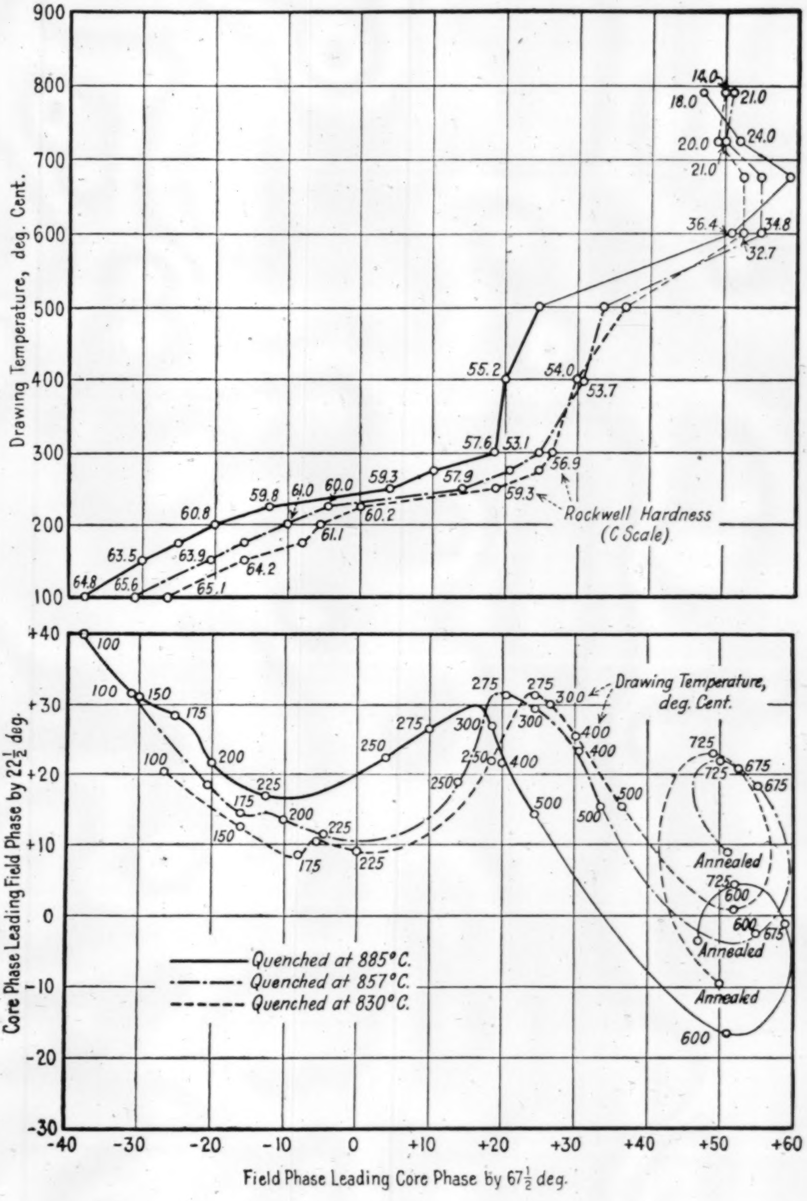

FIG. 7.—Results of Tests with 67½-deg. Lead and 22½-deg. Lag of Galvanometer Phase.

would permit plotting the relative magnetic values of rings having different heat treatments in a two-phase coordinate system.

By shifting the phases to give maximum and minimum readings quite good results could be obtained, but it was only after exploring the effect of phase shifting from a galvanometer field leading the magnetizing core of the tester by 90 deg. to a galvanometer field lagging by 90 deg. that a clearer picture of the test method was obtained.

The arrangement finally adopted is shown in Fig. 5, in which condensers of different capacity are used to change the phase of the galvanometer field current. In choosing the standard it is convenient to use one which will give readings on one side of zero for rings of acceptable hardness and on the other side for rejected rings. A change in the standard will correspond to a change in zero position.

Figure 6 was plotted by taking readings of differently heat-treated rings made from the same bar, and selecting different displacement of core phase to field phase. It is evidently possible to choose a phase relation which will give parallel readings on galvanometer scale and Rockwell hardness, or corresponding physical properties.

By taking readings on a ring with two or three phase relations an approximate estimate of both quenching and tempering can be made. By using two phases having a relation of 90 deg., as shown in Fig. 7, it has been possible to show both the changes in properties taking place by drawing at about 302 to 347° F. (150 to 175° C.) and at about 527 to 572° F. (275 to 300° C.), which intervals correspond to changes in structure, volume, hardness and electric resistance.[1]

By selecting that phase relation between core and field which would give the largest difference in galvanometer deflection for rings of standard heat treatment, and by using as standard a ring of the same size as the rings examined, and with about minimum acceptable hardness limit, easy separation between electrically harder and softer rings than this standard could be obtained in a single reading. The accuracy of the galvanometer reading upon repetition of test is about ± 1 scale division with the primary voltage varying from 110 to 120 volts.

The apparatus could be used for a variety of ring sizes, and with a new Leeds & Northrup quick-acting damped coil, a reading could be made in a few seconds.

Acknowledgment.—The credit for developing the details of the various aspects of this subject should, in chronological order, go to the author's associates in the laboratory, Messrs. Sjövall, Heindlhofer, Dipper and Wefring.

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[1] *Transactions*, Am. Soc. Steel Treating, 1925, p. 34.

DISCUSSION ON MAGNETIC ANALYSIS

Mr. Fahy. MR. FRANK P. FAHY[1] (*Chairman of Committee A-8*).—The principal survey of these bars was carried out by Mr. Spooner. From time to time during the course of Mr. Spooner's work, and subsequent to its completion, different lots of the material were sent to other members of the committee for their investigation. Among those who examined the specimens was Mr. A. V. de Forest, who carried out a large number of tests in which he employed a method which he originated some time ago. I shall now ask Mr. de Forest to present and discuss the results that he obtained.

Mr. de Forest. MR. A. V. DE FOREST[2] (*presented in written form*).—This contribution to the work of Committee A-8 was carried out on the material covered in Mr. Spooner's paper. The method of test is the same as that used on the high-speed drills and reported to the Society in 1923 and described in previous publications.[3] Essentially the measurement is a differential one, quite similar to that described on page 128 of Mr. Spooner's report, but operated in this case with solenoid magnetization at moderate values of induction. The particular feature of the test is the selection of a phase angle at which to operate the alternating current galvanometer which gives the greatest separation of the samples in terms of their physical characteristics, rather than the phase angle which gives a measurement of a definite magnetic constant.

The bars were tested as received and after normalizing, and the results disclosed that one bar cut into the K series was of different character from the others. This difference was found by Mr. Spooner to be in the composition, a 14-per-cent tungsten bar having accidentally found its way into the 18-per-cent material. The magnetic differences among the remaining specimens did not survive heat treatment, and are evidently of secondary importance.

Tests of hardened bars were made using 6-in. solenoids and 1800 ampere turns. The diagram in the accompanying Fig. 1 represents the results of plotting two magnetic readings against each other.

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[1] Consulting Engineer, New York City.

[2] Research Engineer, American Chain Co., Inc., Bridgeport, Conn.

[3] A. V. de Forest, "A New Method of Magnetic Inspection," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 611 (1923); also A. V. de Forest, "Magnetic Determination of the Elastic State," *Transactions*, Am. Soc. Steel Treating, Vol. 6, p. 653 (1924).

Mr. de Forest. These measurements were made at the same magnetizing force but at different phase angles of the galvanometer field and moving coil. The Y-axis is in this case close to the loss axis measured by Spooner and shown in Fig. 14 of his paper; the same specimen, D-6, which lies outside the group of 2350° F. (1288° C.) quench is also out of place in the plot, as are two low reading specimens of graph 2314° F. (1269° C.). The "K" bars are badly out of line, as is also B-2.

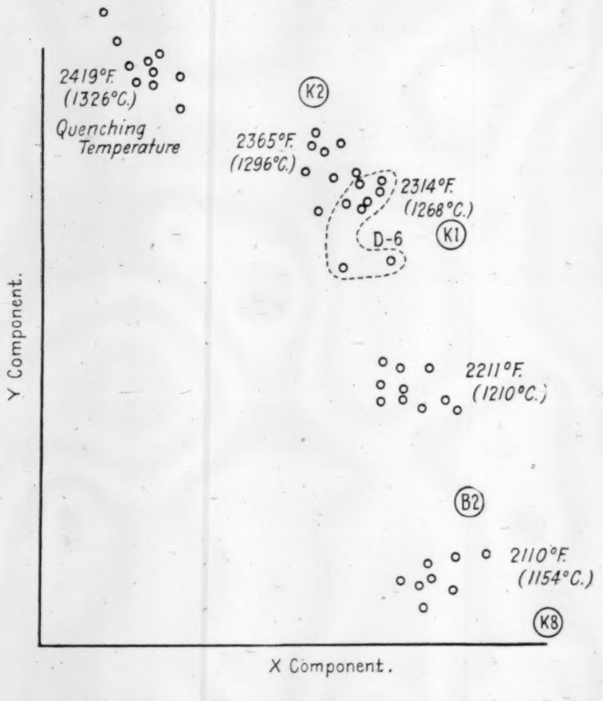

FIG. 1.—De Forest Diagram of Hardened Samples at Medium Induction. 6-in. Coils, 1800 Ampere Turns.

Figure 2 shows the result of measurements on the bars held for various times at high heat. It appears that the one-minute bars have about the same characteristics as those quenched from a lower temperature; while those held for the longer periods than five minutes are quite out of line. This may be due in part to decarburization but is also a question of grain size. The water-quenched bars are entirely away from the main group, showing quite different mechanical characteristics.

One of the interesting possibilities afforded by the double magnetic reading and method of plotting two variables against each other

Mr. de Forest.

is the discrimination after a heat treatment involving both quench and draw between the results of both. If the axes are arranged so that the effect of draw is to displace the position of the spot away from the line of various quenching temperatures, in certain cases the effect of both quench and draw can be measured after complete heat treatment. In order to study the significance of the magnetic readings on the quenched-and-drawn specimens, tests were made of several of the hardened specimens held for various times at high temperature. These were drawn to successively higher temperatures and tested after each draw. An electrically heated salt bath was used, and for the temperatures below 1130° F. (610° C.) a time of only 15 minutes was used. The effect of cumulative draw is probably different from that of a single temperature treatment, so that strict comparison between the bars drawn in this manner and the regular ones is not warranted. Figure 3 shows the variation caused by the drawing treatments, and illustrates the selection of the magnetic properties which are of most use for graphic representation. In this case a phase relation between the field of the alternating current galvanometer and the test circuit was arbitrarily selected such that the water-quenched specimen H-6, and the regular specimen J-2 gave the same measurement and hence lie on a line parallel to the

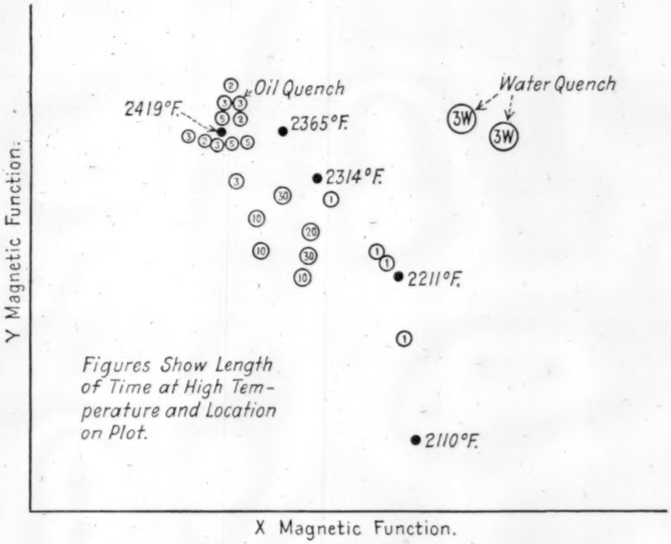

FIG. 2.—De Forest Diagram of Bars Held at High Temperature, About 2350° F. (1288° C.) for Various Lengths of Time. Regular Series as in Fig. 1.

Mr. de Forest. X-axis. Another phase relationship was selected at which the specimens drawn to 1000° F. (538° C.) would read approximately the same as before drawing, and are plotted on a line parallel to the Y-axis. As a matter of time saving the plot is prepared directly from the galvanometer without any numerical readings.

It is seen from Fig. 3 that the progressive drawings move the positions for the three specimens J-2, X-10, and H-6 along lines approximately parallel to each other, and that for each drawing temperature the three read about alike on the Y-axis. It would seem as though the effect of the draw was the same in each case. Compare this behavior with that of J-7, a specimen held 30 minutes at high temperature, and A-5, held one minute. Here, although these read alike on the Y-axis at the start, after drawing to 1000° F. (538° C.) J-7 is far in advance, in the direction of higher draw, than A-5, which has been changed comparatively little. Due to quenching conditions J-7 is in a position to respond more readily to later heat treatment than A-5. The three "regulars" J-2, X-10, and

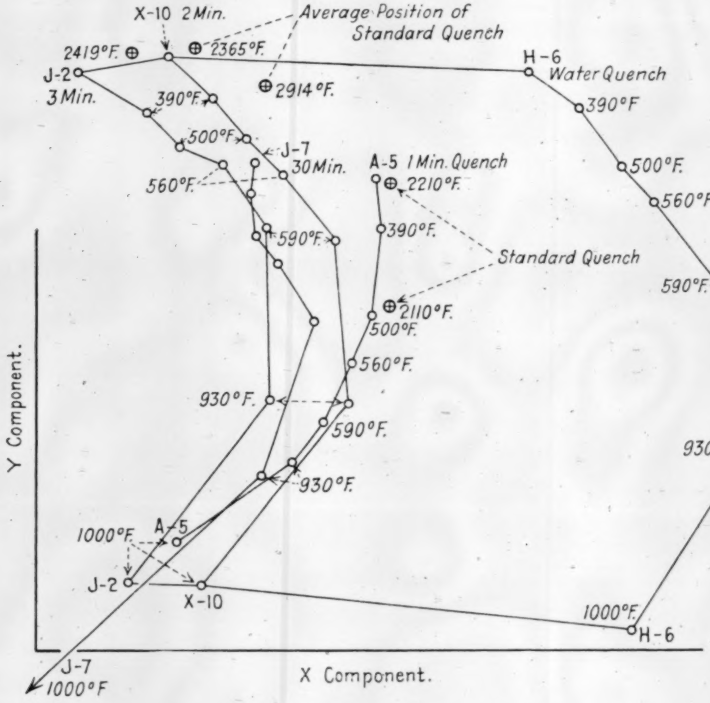

FIG. 3.—Diagram Showing Effect of Drawing Temperature on Bars Held at High Temperature for Varying Lengths of Time.

Mr. de Forest.

H-6 belong to a different category in regard to response than A-5 and J-7, and are intermediate between these latter. Therefore, our plot while covering the two variables of quenching condition and draw for samples J-2, X-10, and H-6 is confused in the case of A-5 and J-7, which latter bring in a third variable. If the plot be considered as the projection on the X-Y plane of graphs which involve three dimensions, A-5 and J-7 lie in space above and below the paper and do not move parallel to each other in the Z-plane, while J-2, X-10, and H-6 can be represented as lying in the plane of the paper, and are therefore in determinate relation to each other. A complete study involving three dimensions has not been attempted here, but would represent the next step in a more complete correlation between mechanical and magnetic properties. The difficulty is more with the mechanical than the magnetic results for the latter are far more precise and sensitive than any chemical, microscopic or mechanical tests so far used to study the behavior of steel.

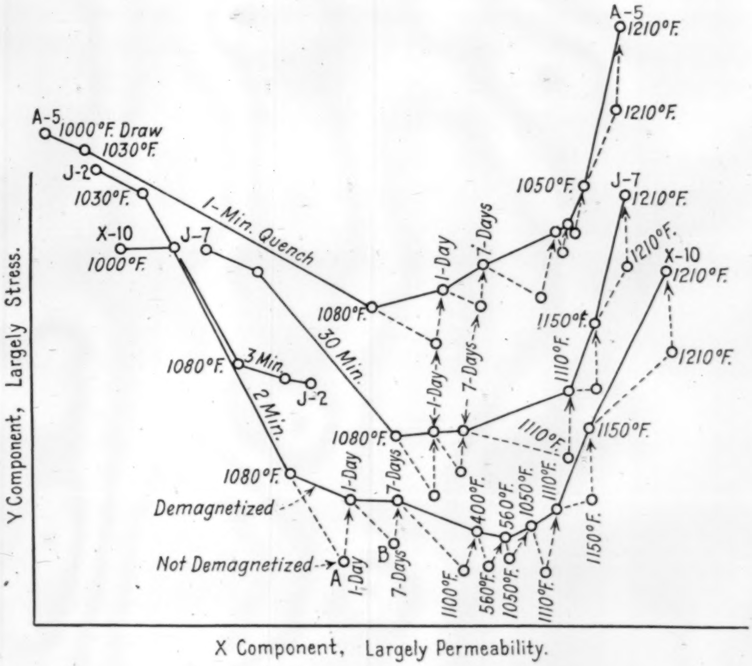

FIG. 4.—De Forest Diagram Showing Effect of Cumulative Drawings and Aging on Various Bars. Continuation of Fig. 3. 6-in. Coils, 800 Ampere Turns.

Figure 4 is a continuation of Fig. 3, showing the effects of higher drawing temperatures, and changes due to aging and reheating.

Mr. de Forest. The curve for X-10 is given in greatest detail and will, therefore, be described. The phase relations have been shifted to give the best separation of the results, and readings on the X-axis are proportional to permeability while Y is at a small but important angle from the loss axis. The positions on the plot at 1000° F. (538° C.) draw represent the same condition as at the last station in Fig. 3. H-6, water quenched, is off scale at the sensitivity used for the Y-axis. A 15-minute draw in the salt bath caused the change to the point marked 1030° F. (554° C.); further drawing to 1080° F. (582° C.) brought the bar to that position. So far all the results plotted have been obtained after demagnetization with a sufficiently high induction at 60 cycles. Previous observation has always shown a shift in position due to this demagnetization, which seems to be more of a magnetic-stress phenomenon than a demagnetization in the usual sense. It might be considered that the effect of dimensional change due to magneto-striction exerted a small stress cycle throughout the mass of metal. This stress cycle would cause slip to take place along the most highly strained slip planes, and would produce a somewhat diminished state of internal stress. The result would be a permanent change in magnetic properties after any heat treatment and this change ought to be always in a direction of less stress, and in general toward higher drawing temperatures, which usually result in greater mobility and decrease in internal stress. In high-speed steel, martensite is known to be formed from the austenite retained by the quenching treatment. This transformation results in a change in volume and consequently in a high state of internal stress; perhaps through the mechanism pictured by Bain and St. John.[1] The temperature of maximum formation of martensite would correspond with the red hardness temperature of draw, in this case about 1050 to 1100° F. (566 to 593° C.). The demagnetization, if it is a destressifying mechanism, should act in the same way below and above that temperature, but below that temperature further drawing would be partly in the direction of more stress, and above that temperature in the direction of less stress. The shape of the curves in Fig. 4, for instance that of X-10 from 1000 to 1210° F. (538 to 654° C.) draw, might be due to the increase of stress by heat treatment below 1080° F. (582° C.), and to the decrease beyond 1110° F. (599° C.). If this assumption that the Y-component is mostly a matter of stress is correct, the demagnetizing effect should always be to shift the point upward, toward less stress instead of in the direction of further draw. An inspection of the plot shows that this is borne out extremely

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[1] E. C. Bain, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., February, 1924.

Mr. de Forest.

clearly. The points below the line, connected by dashes to the demagnetized curve, lie directly below the corresponding points, regardless of the fact that at 1080° F. (582° C.) the next step slopes downward, and at 1150° F. (621° C.) upward.

The position on X-10 marked 1080° F. (582° C.) was reached by a 15-minute draw in a salt bath. The measurement was made after the usual demagnetization. Twenty-four hours later the measurement was repeated, first without demagnetization, giving point marked "A." Demagnetizing shifted this position to that on the curve above it marked "1 day." Thus the aging effect produced a change to the right accompanied by a growth of internal stress, some of which is permanently associated with the change in structure, but much of which is removed by the demagnetizing treatment. Further aging for a week produced a change to "B," the demagnetized value of which is at "7 days," above. Further aging was not practical, so reheatings to lower temperatures were used to determine whether equilibrium continued toward the production of stress and hence presumably toward martensite, or away from it. An hour at 400° F. (204° C.) gave the results shown, a change from "7 days" to the lower "400° F." nearly parallel to the aging change.

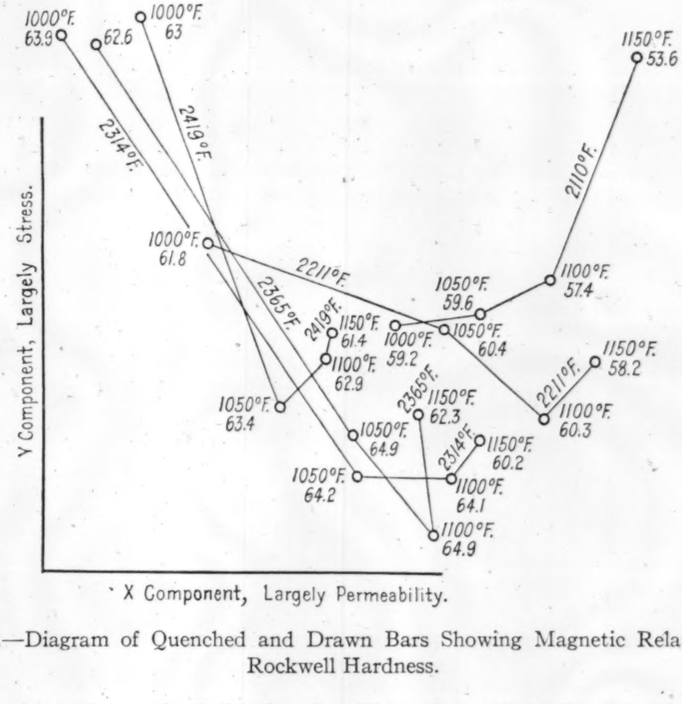

FIG. 5.—Diagram of Quenched and Drawn Bars Showing Magnetic Relation to Rockwell Hardness.

Mr. de Forest. This reheat evidently builds up a stress about proportional to the change in structure; in other words is more of the same thing as the aging. At the next point, 560° F. (293° C.) reheat, there is a turn toward a permanent lessening of stress, but reheat still builds it up, presumably from precipitation of more martensite. At temperature 1110° F. (599° C.), however, a profound change takes place. The draw to 1150° F. (621° C.) instead of building up stress, lessens it, and point 1150° F. (621° C.) as drawn lies above and to the right of 1110° F. (599° C.) demagnetized. The added mobility due to the draw then allows a further permanent lowering of stress shown by the change from 1150° F. (621° C.) as drawn to 1150° F. (621° C.) demagnetized. Further draw from 1150 to 1210° F. (621 to 654° C.) has a similar effect, only more marked. There is a larger amount of stress released by the heat treatment above, and equilibrium is going toward a lessening of stress rather than toward an increase as at point 1080° F. (582° C.).

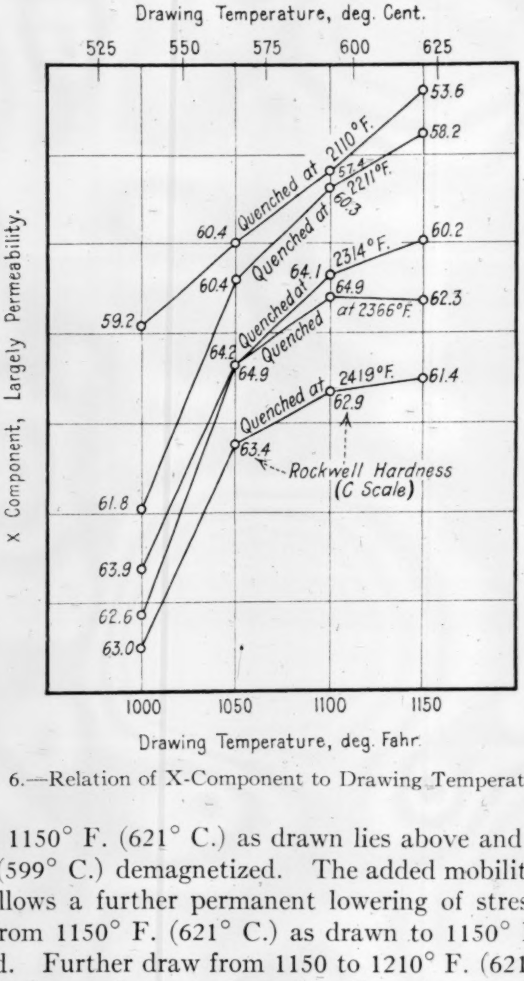

FIG. 6.—Relation of X-Component to Drawing Temperature.

Mr. de Forest.

The curves for one-minute quench, A-5, and 30-minute quench, J-7, lie above X-10, evidently reflecting the fact that neither have been prepared by their quenching treatment for the development of a maximum internal stress, and perhaps also of martensite. The course of the curves for A-5 and J-7 indicate that the conditions of maximum stress occur at a lower drawing temperature than for X-10. As far as A-5 is concerned this conclusion seems well supported by evidence,[1] that lower temperatures of quench give their maximum hardness at lower temperatures of draw.

With the above discussion in mind it is possible to interpret more freely the rather complicated picture presented by the complete set of quenched-and-drawn bars as prepared by Spooner. Figure 5 shows these results, plotted to the same axes as used in Fig. 4, with stress along the Y-axis; in both cases the X-coordinate is approximately equivalent to permeability. If these latter values are plotted as in Fig. 6, there is a striking resemblence to the results shown by Spooner in his Fig. 21. In fact similar relations between the bars are shown by tests at quarter the induction used in the case illustrated as well as at 20 times that induction. Other tests indicate that frequency changes from 45 to 90 cycles, and various wave forms, have also negligible effects. Whatever mechanical properties are here reflected, they seem to offer a most convenient opportunity for magnetic inspection when but one variable is to be dealt with. With two magnetic measurements a more general view of the situation may be obtained, and both in research on the metallography of steel and in routine inspection of commercial products, a check can be made against the possibility that some assumption, such as uniform chemical composition or quenching treatment, may sometimes be wrong.

The picture presented by Fig. 5 suggests this interpretation in the light of the argument on Fig. 4. The dip in each curve corresponds to the position of maximum secondary hardness, percentage of martensite, internal stress, and probably brittleness as well. There is a particular high heat treatment which allows this maximum to develop to the greatest extent; both 2314 and 2419° F. (1268 and 1326° C.) fail to reach the result of 2365° F. (1296° C.) quench. However, this does not mean that only the temperature and time at high heat cause this result.

There is also a variation in individual response due to all the factors of composition and treatment represented in the metal at the time of quench, and this initial difference may in many cases prevent

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[1] M. A. Grossman, "Shrinkage and Expansion of High-Speed Steel Due to Heat Treatment," *Chemical and Metallurgical Engineering*, September 13, 1922.

Mr. de Forest. a rigorously accurate statement of the exact quenching temperature which has led to the final position on the plot. Indeed it is only the mechanical properties of the finished bar which are of immediate interest, and these unfortunately are difficult to determine. The easiest of these factors to measure, hardness, was run on a Rockwell machine C scale and the figures on Fig. 5 give the average of ten readings on each bar. Here it is evident that although hardness varies little in the secondary hardness range, there is a marked agreement between these figures and the shape of the magnetic plot. A similar result was shown in a previous paper.[1] A striking feature of this diagram is the sharpness of the reversal in the Y-coordinate, in the case of 2365° F. (1296° C.) and the 2419° F. (1326° C.) quench series especially. Whether a greater number of test specimens would bear this out would be a subject for further test. It would likewise be interesting and perhaps profitable to deduce from the magnetic reading after quench, what drawing temperature would result in the greatest secondary hardness.

Conclusions:

1. It is shown that magnetic tests can be made which give a possible explanation of the changes going on during the drawing process of quenched high-speed steel.

2. A simple form of test circuit operated on 60-cycle commercial current, with moderate values of induction and solenoid magnetization, can yield the same results as are obtained at high induction with the use of a yoke. This check indicates that a simple form of comparison can be depended upon even under widely differing conditions of method and apparatus.

3. Either a double or single test may be adjusted to read directly in terms of variables represented in the material used, rather than in terms of purely magnetic constants.

4. A double reading test in many cases can be made on quenched-and-drawn material and can furnish a check on the correct effect of both operations.

5. A laboratory study of the magnetic and mechanical factors can best be made, using a double test; and if conditions are then found such that a single test is sufficient for inspection, the most favorable magnetic circuit can easily be arranged.

Mr. Fahy. MR. FAHY.—Now that Mr. Spooner and Mr. de Forest have presented the story of the magnetic variations corresponding to the different heat treatments, it will be of interest to have Mr. Styri tell us what is disclosed in his examination of the specimens under the microscope and in testing for hardness. **Mr. Fahy.**

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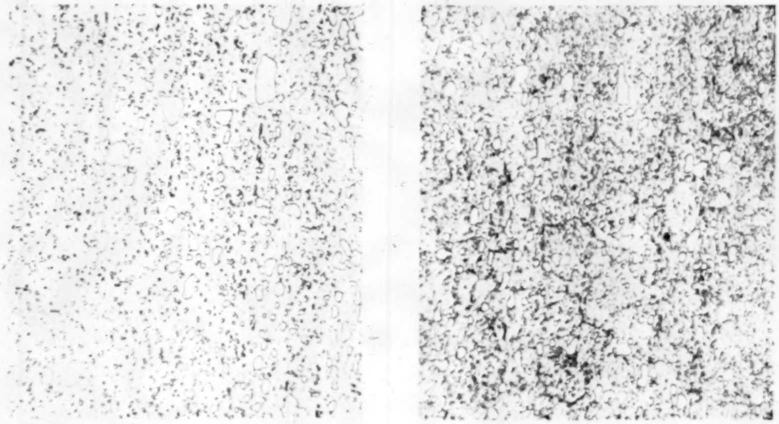
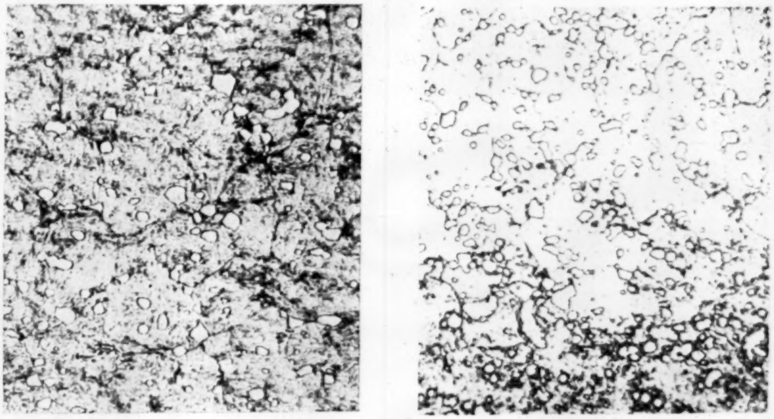
[1] A. V. de Forest, "A New Method of Magnetic Inspection," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 611 (1923).

Mr. Styri. MR. HAAKON STYRI (*presented in written form*).—The photomicrographs reproduced in Figs. 15 to 19 of the paper and in the



Specimen G-5, Quenched from 2100° F. (1150° C.), Drawn at 1000° F. (540° C.)

Specimen L-6, Quenched from 2300° F. (1260° C.), Drawn at 1000° F. (540° C.)

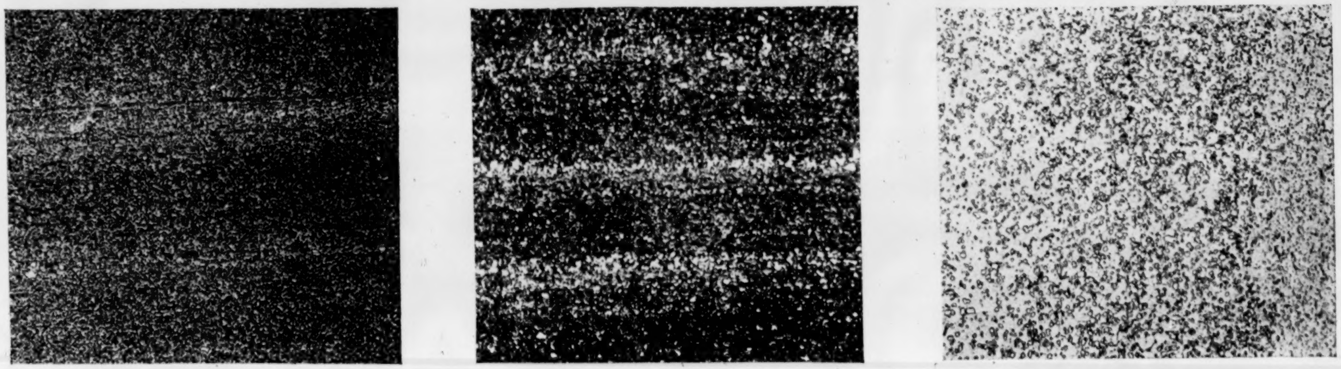


Specimen X-7, Quenched from 2400° F. (1315° C.)

Specimen A-6

FIG. 7.—Photomicrographs of High-Speed Steel (× 500).

accompanying Figs. 7 to 9 were taken on a side surface after grinding down about $\frac{1}{16}$ in. and preparing carefully. Specimens K-10 and G-4 represent the structure of the normalized bars at a magnification of 500. Specimens B-2, B-10, and I-5 represent the structures of hardened but untempered bars at magnifications of 500 and 2000

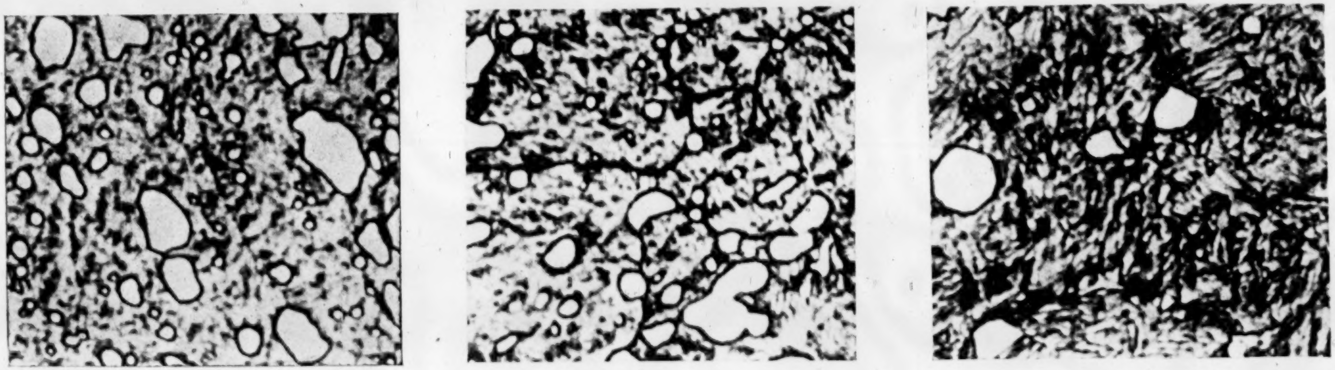


Specimen G-5, Quenched from 2100° F. (1150° C. Drawn at 1000° F. (540° C.)

Specimen G-2, Quenched from 2400° F. (1315° C.)

Specimen A-6

FIG. 8.—Photomicrographs of High-Speed Steel (× 100).



Specimen G-5, Quenched from 2100° F. (1150° C.), Drawn at 1000° F. (540° C.)

Specimen L-6, Quenched from 2300° F. (1260° C.), Drawn at 1000° F. (540° C.)

Specimen X-7, Quenched from 2400° F. (1315° C.)

FIG. 9.—Photomicrographs of High-Speed Steel (× 2000).

Mr. Styri.

and respectively for 2100, 2300 and 2400° F. (1150, 1260 and 1315° C.) quench; specimen K-1 the structure of bars quenched from 2350° F. (1290° C.), and specimens G-5, L-6 and X-7 represent structures of hardened and tempered bars at magnifications of 500 and 2000 respectively for a quench of 2100° F. (1150° C.) drawn at 1000° F. (540° C.), for a quench of 2300° F. (1260° C.) drawn at 1000° F. (540° C.) and for quench of 2400° F. (1315° C.).

The sections shown have a more uniform structure than a length-section through the center of the bars or a cross-section would have. The specimens G-5 and G-2 at a magnification of 100 respectively for a quench of 2100° F. (1150° C.) drawn at 1000° F. (540° C.) and for a quench of 2400° F. (1315° C.), show this greater streakiness in the center of the bar. Practically all the bars show this streakiness which was particularly prominent in A-1. The higher quenching temperature will produce more contrast in the streaks.

Specimens which have been heat treated alike show no noticeable difference under the microscope when examined in cross-section and length-section. (Any difference of the photomicrographs is more or less accidental, as they were taken in a routine manner without any previous complete study of structure.) When the groups are compared with each other, however, some variations with heat treatment are rather evident.

The grain boundaries, which are most easily developed by etching in 1-per-cent HNO_3 in alcohol, appear much sharper in the untempered than in the drawn specimens. It was of some interest to find at what degree of drawing this change took place, and cubes from specimens A-1 and A-3 were examined after heating in salt baths for one-half hour at various temperatures, up to 1100° F. (595° C.) The grain boundaries in the A-1 specimens (quenched at 2100° F. (1150° C.) would become indistinct after 1000° F. (540° C.) and hardly visible after 1100° F. (595° C.) even when examined at a magnification of 1000 with oil-immersion objective which would usually permit observation of the grain boundaries much better than examination at magnification of 500. In the A-3 specimens (quenching at about 2350° F. (1290° C.), the grain boundaries are more persistent, but become indistinct after tempering at 1100° F. (595° C.).

In general, the grain boundaries are more easily developed with increase in quenching temperature. There is also a corresponding increase in grain size, particularly noticeable after quenching at 2350 and 2400° F. (1290 and 1315° C.). It takes longer to develop the structure within the grains when the quenching temperature has

Mr. Styri. been high, and the Kourbatoff reagent or a solution of 1-per-cent HCl in alcohol are both satisfactory for this purpose.

There are fewer carbides present after higher quench and the martensitic structure becomes coarser and sharper. At about 2100 to 2200° F. (1150 to 1200° C.) quench the martensite in indefinite and much like that of high-carbon chrome steel quenched at about 1525° F. (830° C.) in oil, while a quench at 2350 to 2400° F. (1290 to 1315° C.) gives a coarser martensite like that of carbon-chromium steel quenched in oil at 1575 to 1600° F. (855 to 870° C.).

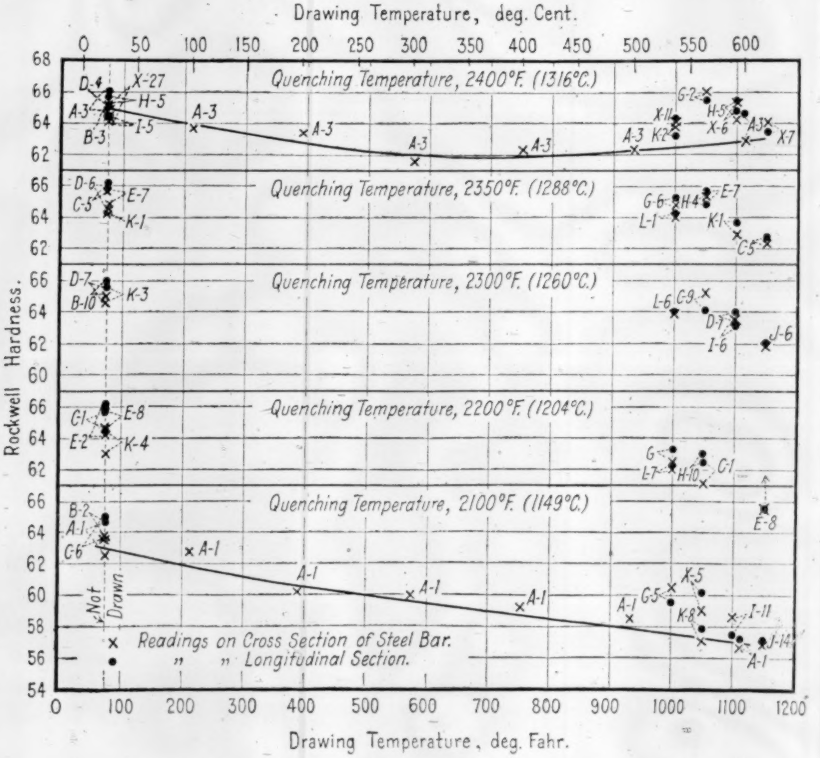


FIG. 10.—Results of Rockwell Hardness Tests on High-Speed Tool Steel of Various Heat Treatments.

After drawing, the etching time will be less, starting from about 750° F. (400° C.) and for high quenches and draw the acicular structure will have more contrast.

The A-6 specimen is interesting in another respect, namely, in having a very decided decarburization of the surface extending to about 0.030 in. and produced by the excessive time in the salt bath.

Mr. Styri. The surface grains are also much larger than the inside grains, and the carbides are thinning out towards the surface as shown. The specimen at a magnification of 500 shows the zone of the end of decarburization, when the martensite becomes distinct.

The K specimens can hardly be distinguished from the other samples of similar heat treatment.

For estimation of hardness, Rockwell tests were made at three or more points on the prepared side surfaces and also on the cross-sections and on some of the center length-sections. The average results are given graphically in Fig. 10 and show that the readings on the faces of most of the untempered specimens and on some of the tempered ones usually are higher than the readings on the cross-sections. When a little more is ground off the face or if the hardness of a length-section through the center is taken there is less difference. This is also true for the A-1 and the A-3 specimens which were drawn after the cubes were cut from the bar and is therefore probably due to the original quench and has perhaps been influenced by insufficient soaking at the drawing temperature.

The specimens quenched at low temperature show a decided drop in hardness with increased degree of tempering, but the samples quenched at high temperature maintain their hardness after drawing, except perhaps for a possible drop at some intermediate drawing at about 800° F. (425° C.), indicating a secondary hardening.

The lines drawn for the A specimens are not intended to show a smooth variation in hardness with increased drawing, but to show the general tendency.

In conclusion, it can be said that the variations in microstructure and Rockwell hardness indicate undesirable non-uniformity of the original material for a test of this nature and also that differences have been introduced in the heat treatment, particularly by insufficient soaking time at quenching heat.

Mr. Fahy. MR. FAHY.—In calling next upon Mr. R. L. Sanford, I would stress the point that there are large magnetic differences between the K specimens and the others, although Mr. Styri's work shows no difference between them in grain size and Rockwell hardness. We cannot be entirely certain at this time of the actual differences which would be disclosed when the material is shaped into a cutting tool, but practical experience would lead us to expect a corresponding variation in performance, other things being equal.

Mr. Sanford. MR. R. L. SANFORD.[1]—One of the great advantages of a cooperative investigation such as the one under consideration is that, without unnecessary duplication of effort, the problem can be simul-

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[1] Chief, Magnetic Section, U. S. Bureau of Standards.

Mr. Sanford. taneously attacked from a number of different points of view. At the same time, general conclusions which follow from the results obtained independently by two or more investigators are strengthened by such corroborative evidence.

It is the opinion of the writer that the surest, although not necessarily the most rapid progress in magnetic analysis, will be made by investigations of a fundamental and more or less theoretical nature. To be explicit, it is desirable to determine the magnetic properties of the various elements and compounds which enter into the composition of the materials under investigation and the way in which these individual components, in the various combinations which may exist, contribute to the resultant magnetic properties which we are able to measure. It is realized that this is a tedious and difficult method of procedure, but in the light of the results of many investigations, carried out from other points of view, this seems to be the best way of ascertaining the facts and laying a secure foundation on which to build up the practical application of magnetic analysis. It might also be pointed out that high-speed steel with its complex composition and complicated constitution offers as difficult a subject for this type of study as might well be imagined.

The work of the Bureau of Standards in magnetic analysis during the past few years has, in so far as possible, been directed along the line of such fundamental research. We are in entire accord with the conclusion of Mr. Spooner that it is only by working at the higher inductions that we can avoid the confusing effects of mechanical strain and possibly of other factors which exert a greater influence on the magnetic properties than on the mechanical properties with which we wish to correlate them. It would be of great advantage if there were some definite law by which the properties of the material could be defined in terms of constants which could be experimentally determined. The so-called Kennelly law or reluctivity relationship has been suggested in this connection and the evidence thus far obtained seems to indicate that it is valid and can be used in the analysis and interpretation of the results of magnetic tests at high inductions. In accordance with this law, which in general holds from a point somewhat above maximum permeability upwards, the graph obtained by plotting ferric reluctivity (the reciprocal of permeability) against the magnetizing force is a straight line for pure and magnetically homogeneous materials. This straight line has two characteristics which may properly be called magnetic constants because they do not depend upon the degree of magnetization. They are the slope and the intercept on the axis of reluctivity. The reciprocal of

Mr. Sanford. the slope is numerically equal to the magnetic saturation value. If there is more than one magnetic component present, each component follows its own characteristic line and the resultant is not straight but curved. If it were possible to resolve an experimentally determined reluctivity curve into its straight line components, we would have a method of determining the magnetic characteristics of the distinct magnetic components of which the material is composed. There are two phases of this line of attack which require considerable study. One is the improvement of testing methods so as to obtain experimental data of the required accuracy and the other is the problem of the mathematical analysis of the results. Each of these phases of the problem presents some difficulties, but it does not appear that they should be insuperable.

TABLE I.—MAGNETIC SATURATION VALUE AND COERCIVE FORCE.

Sample Number	Quenching Temperature		Drawing Temperature		Saturation (Gausses)	Coercive Force[1]
	deg. Fahr.	deg. Cent.	deg. Fahr.	deg. Cent.		
A-1	2109	1154	No draw	...	15350	58
L-11	....	....	1000	538	16700	42
X-5	....	....	1050	566	.....	..
X-23	....	....	1100	593	16050	38
X-24	....	....	1150	621	16500	38
K-4	2211	1211	No draw	...	.....	..
L-7	....	....	1000	538	16130	46
X-1	....	....	1050	566	16580	40
X-20	....	....	1100	593	16500	40
X-26	....	....	1150	621	16750	40
K-3	2316	1269	No draw	...	.....	..
L-6	....	....	1000	538	.....	..
X-14	....	....	1050	566	16530	42
X-16	....	....	1100	593	17200	44
X-22	....	....	1150	621	16900	42
A-4	2366	1296	No draw	...	13930	62
L-1	....	....	1000	538	15510	46
X-6	....	....	1050	566	16620	42
X-12	....	....	1100	593	17350	36
X-13	....	....	1150	621	16660	42
X-9	2419	1326	No draw	...	14770	64
X-11	....	....	1000	538	.....	..
X-15	....	....	1050	566	17030	48
X-17	....	....	1100	593	16950	42
X-19	....	....	1150	621	16950	48

[1] Hmax = 1000 gilberts per centimeter.

The regular apparatus used at the Bureau of Standards for magnetic measurements at high inductions is not adapted to specimens of the size used in the present investigation. An improvised set-up was tried out which appeared to give good results but further study revealed a source of error which rendered the values too inaccurate for the application of the reluctivity relationship. This was not discovered until the bars had received the second heat treatment. Measurements have been made by means of the Fahy Simplex per-

Mr. Sanford. meameter with the high magnetization adapter on a number of the bars after the drawing treatment. Some of the bars had previously been sent to other laboratories for test so that the information is not complete. The type of results obtained is indicated in Table I and the reluctivity curves of Fig. 11. None of the reluctivity lines were straight and the saturation values obtained from them were calculated in terms of the slope of the asymptote of the curve. Values of coercive force measured from a maximum magnetizing force of 1000 gilberts per centimeter are also given in the table. These values are given as a matter of information as time did not permit further attempts at analysis.

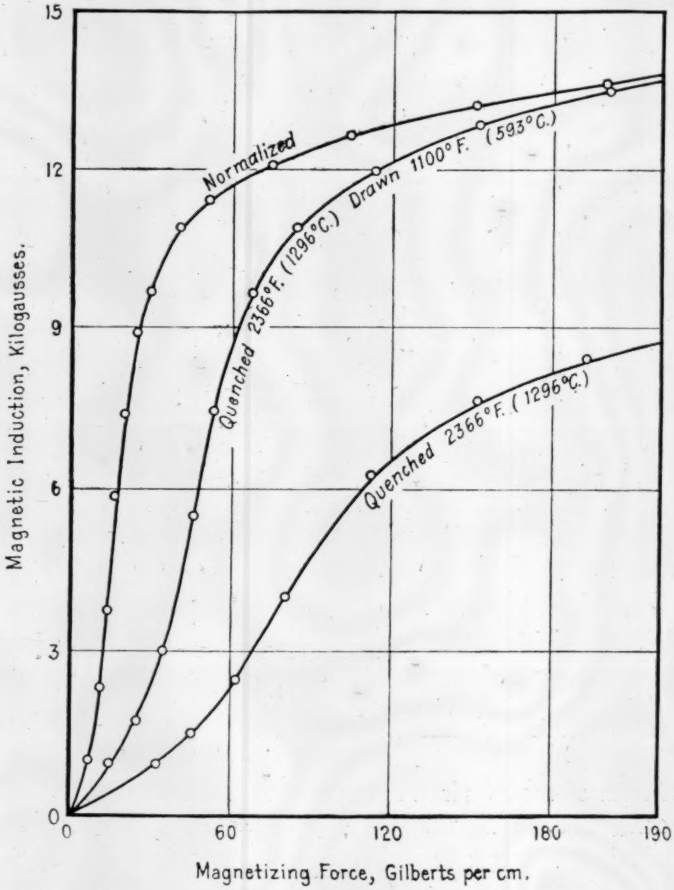

FIG. 11.—Magnetic Reluctivity of High-Speed Steel Quenched at 2366° F. (1296° C.).

In Fig. 12 are given curves of normal induction for magnetizing

Mr. Sanford.

forces up to 300 gilberts per centimeter for typical bars in the normalized, quenched, and quenched-and-drawn conditions. These curves illustrate the magnitude of the change in magnetic properties brought about by the heat treatment.

Mr. Fahy.

MR. FAHY.—Mr. Sanford, in referring to Fig. 12, points out that his derived curve indicates a possible lack of uniformity, presumably resulting from differences in metallographic constituents across the section. Mr. Styri has pointed out that the material was found to be "streaky." I might say that the committee desired to work with the usual run of material such as would normally be encountered in tool-manufacturing operations.

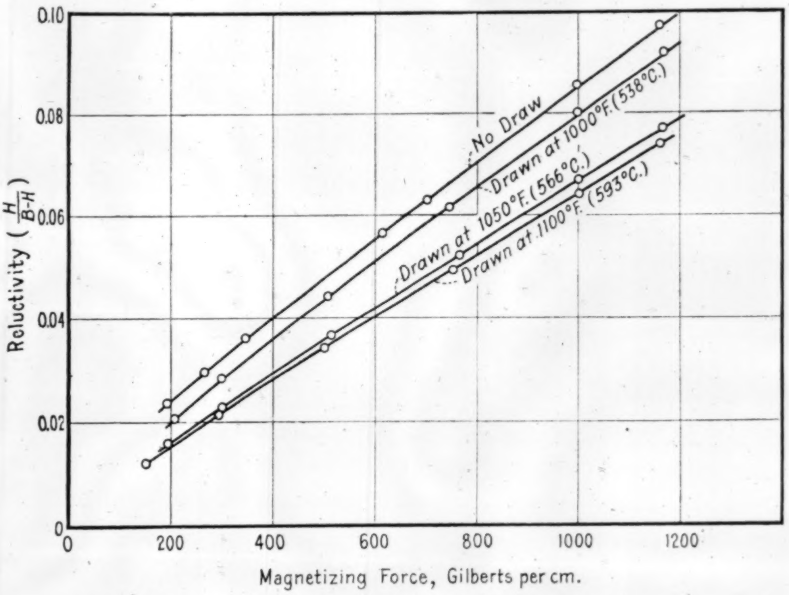

FIG. 12.—Normal Induction of High-Speed Steel.

I will now ask Mr. M. A. Grossman, who has some acquaintance with the general test program and is very familiar with high-speed steel in practice, to give us the benefit of his comments.

Mr. Grossman.

MR. M. A. GROSSMAN.[1]—It seems to me quite fitting first to offer a few words of appreciation of the painstaking work of the various members of the committee: it is totally new and promises to be a very easy method of inspection. Probably the outstanding benefit that will come from it is that it offers a method for determining previous heat treatment in two dimensions. We have had, for

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[1] Metallurgical Engineer, United Alloy Steel Corporation, Canton, Ohio.

Mr. Grossmann. example, hardness, which will not determine both the quenching and the drawing temperatures. We know that if high-speed steel has been quenched, and is drawn at successively higher temperatures, it first softens, becomes slightly harder again, and finally softens; so that any particular Rockwell hardness might represent one of several particular quenching temperatures over a given range. The same thing applies to density measurements, because a high-speed steel which has been quenched, first contracts, then expands and then again contracts. There is, perhaps, a way of estimating roughly what a heat treatment has been by observation of the microstructure, because, as the quenching temperature increases, the grain size increases, and as the drawing temperature is raised, there is a change in the structural appearance within the grains. This structural appearance, taken in conjunction with the time required for etching, sometimes gives a fairly accurate index of the previous heat treatment. However, with the magnetic method of inspection, particularly as developed by Mr. de Forest and Mr. Styri, you will remember that two magnetic properties are plotted against each other, and the curves are distinctly separate for the different quenching and drawing temperatures. I was particularly interested in Mr. de Forest's figures in which the curve for a quenching temperature of about 2350° F. (1290° C.) and a drawing temperature of about 1100° F. (595° C.) showed a marked maximum. This point, as we all know, represents the best heat treatment for high cutting efficiency in the ordinary 18-per-cent tungsten steels, and probably also represents the maximum degree of irregularity of distribution of constituents in high-speed steel. So far as I am aware, this is certainly the most sensitive method that has been developed for determining the heat treatment of a previously heat-treated piece of high-speed steel.

Mr. St. John. MR. ANCEL ST. JOHN.[1]—The report which has just been presented, with the valuable discussion thereof, merits the serious attention of all makers and users of steel, for it sets forth a practical non-destructive method of securing information concerning the suitability of the pieces examined for the service for which they are intended. The committee will undoubtedly continue the investigation, not only with respect to high-speed steel, but also with respect to other steels. They will also delve much more deeply into the correlation between magnetic tests, other methods of testing, and the properties of the materials tested. But the important fact from the practical point of view is that with respect to the material now under consideration, magnetic testing can tell whether the article examined has had the treatment required and whether it is in a suitable state to be used. **Mr. St. John.**

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[1] Consulting Physicist, New York City.

As I said three years ago of X-ray examination of castings, I now say of magnetic analysis, "It is time to stop asking the question, 'can we afford to use this method?' and to start asking 'can we afford not to?'" Unless I am greatly mistaken a few organizations with sufficient vision to be willing to work out their own inspection problems could within a reasonable time recover the entire cost in the savings effected, and at the same time assist the work of the committee immeasurably. It is significant that at this convention a tentative recommended practice for metal radiography is being put forth. I trust that within another three years similar action will have been taken with respect to magnetic analysis of this sort, and that organizations here represented who are not now doing anything material to further this work will be among the principal sponsors of that action.

Mr. Job. MR. ROBERT JOB.[1]—I should like to ask whether the method of magnetic analysis may be used in detecting blow holes and similar defects in large steel castings of very irregular dimensions?

Mr. Fahy. MR. FAHY.—In answer to Mr. Job, I would reply that this has been attempted but is fraught with considerable difficulty in certain circumstances. Where the cavity is large, that is, where there is a relatively large loss in section, the determination is rather simple; but if the hole is small and especially if it is at a considerable distance from the surface we can only say that the technique has not yet advanced to the required point.

Magnetic analysis is not new. The first recorded attempt to use the magnetic characteristics to determine the quality or state of ferrous materials dates back to about 1850 in this country, and earlier abroad. If we look into the reasons for its slow development we find the outstanding one to be the lack until very recent times of adequate means and methods of magnetic testing. It is only during the past twenty years that we have had really competent means for accurately testing short straight bars. It is only natural that such conditions should retard practical application even when, in isolated instances, satisfactory results were obtained by very crude procedures. With the gradual improvement in the methods of magnetic testing and the design of auxiliary apparatus, a feeling of confidence has grown. Really fundamental work becomes possible. We are now able to explain the causes of some of the apparent anomalies which have greatly exercised and confused some of the early, and in

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[1] Vice-President, Milton Hersey Co., Ltd., Montreal, P. Q., Canada.

Mr. Fahy. fact quite recent, investigators. For example, in the magnetic examination of wire there are often found very substantial magnetic differences which might be expected to indicate the presence of regions having other physical properties than those of the main run of the material. However, laboratory and service tests often fail to show that there are associated inhomogeneities of importance. Mr. R. L. Sanford, a member of this committee, has conclusively shown during the past year that under low magnetizing forces the effects of internal stress play a very large part in determining the magnetic flux strength and that the practical determination of such inhomogeneities as are important from the standpoint of mechanical performance demands that the magnetizing forces employed be considerably above the values commonly used hitherto. This is a very important finding that will greatly affect future work.

For its proper and rapid development, magnetic analysis requires more work of this fundamental order. This the committee is attempting to carry through and at the same time to stimulate interest among those who are so placed that they can assist by making now such practical applications in their own plants as are feasible in the present state of the art. The fact remains that no worker in magnetic analysis has yet gone on record to the effect that he has found two specimens of material which were the same magnetically and yet were substantially different from the service standpoint. We do not know of any other method of test which is quite so sensitive and allows inspection to the same degree without injury to the material.

The committee at a meeting held in conjunction with this annual meeting planned work which it is believed will carry matters well forward during the coming year. It is now to have the cooperation of Mr. St. John, who has very kindly offered his personal assistance as well as the facilities of his laboratory, and his technique in X-ray applications will, it is believed, prove of invaluable assistance in clearing up some outstanding matters.

Committee A-8 welcomes all the help it can get, believing that the results it has obtained and will ultimately obtain will finally prove of benefit to industry in general. It in turn offers such assistance as it can give to any member of the Society who wishes to take up inspection work of this type and points out the likelihood that in many cases a very simple form of magnetic test may prove very valuable as a means of inspecting a finished or semi-finished product.

A NOTE ON THE RELATIONS BETWEEN THE PROPOSED STANDARD TEST BAR FOR CAST IRON AND THE TWO EXISTING STANDARDS

By J. T. MacKenzie[1]

Synopsis

The paper presents results on five cast irons of varying analysis showing the empirical relationships between the load and deflection of the pipe standard bar (2 by 1 by 24 in.), the arbitration bar (1.25 in. in diameter by 12 in.), and the proposed new standard bar (1.2 in. in diameter by 18 in.). The beam formulas are given and the differences between the empirical and theoretical values are shown.

The conversion factors found, using the proposed bar as 100 per cent, are for load and deflection in the order named, 168 and 53 per cent for the arbitration bar and 112 and 162 per cent for the pipe bar.

It is a well-recognized fact that cast iron tested transversely to failure does not follow the conventional beam formulas. Since these formulas have been derived mathematically and apply only to a homogeneous, isotropic, and elastic material, they are approximately true, when applied to cast iron, only for correcting bars deviating slightly from standard dimensions. The results presented in this paper are given to show the actual relationship and the deviation from the elastic formulas.

The three bars investigated are: the standard test bar for cast-iron pips,[2] which is 2 by 1 by 26 in. long tested flatwise on supports 24 in. apart, hereafter referred to as "P"; the arbitration test bar[3] which is 1.25 in. in diameter and 15 in. long tested on supports 12 in. apart, referred to as "A"; and the new bar, proposed by the American Foundrymen's Association to supplant the present arbitration bar, which is practically the old standard of the International Association for Testing Materials and is 1.2 in. in diameter and 20 in. long, tested on supports 18 in. apart. It is hereafter referred to as "N."

Description of Test

Each set consisted of from three to five molds for each size bar all made in the same way, that is, rammed one to the flask with pipe-foundry molding sand, blacked with pipe blacking, thoroughly dried overnight and cast on end mounted on a blacked and dried fire brick. This method gives a bar with a smooth, clean skin, free from sand or scale, and easy to measure. It also gives clean bearing surfaces so there is no crushing of imbedded sand grains to confuse the deflection readings. The sets were all poured in the same order—N, P and A—and all bars of each set were poured from the same ladle.

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[1] Chief Chemist, American Cast-Iron Pipe Co., Birmingham, Ala.

[2] Standard Specifications for Cast-Iron Pipe and Special Castings (A 44 - 04), Section 9, 1924 Book of A.S.T.M. Standards, p. 386.

[3] Standard Specifications for Gray-Iron Castings (A 48 - 18), Section 7, 1924 Book of A.S.T.M. Standards, p. 409.

The base materials for sets Nos. 358, 359, 360, and 366 were all from the same car of pig iron and the same kind of steel scrap, the proportion varying from 50 per cent in No. 358 up to 100 per cent pig iron in No. 360. Set No. 374 was from the customary mixture for special castings. The first four sets mentioned were broken on a Riehlé autographic transverse machine, but in order to check this machine set No. 374 was tested by the Pittsburgh Testing Laboratory on an Olsen universal machine and the results, though complicated by the change in phosphorus, seem to be fairly consistent. Five pipe bars were made in this set, the first and last tested by the author and the middle three by the Pittsburgh Testing Laboratory. The Riehlé machine showed a load of 2390 lb. and 0.37 in. deflection, while the Olsen machine showed a 2440-lb. load and 0.375 in. deflection. The curves were plotted by the Pittsburgh Testing Laboratory from load readings taken at each 0.02 in. of deflection.

Table I.—Chemical Composition of Sets.

Set	Silicon, per cent	Sulfur, per cent	Manganese, per cent	Phosphorus, per cent	Graphitic Carbon, per cent	Total Carbon, per cent	Carbon plus Silicon/3, per cent
No. 358	1.14	0.11	0.39	0.16	1.95	3.20	3.58
No. 359	1.68	0.09	0.53	0.18	2.49	3.30	3.86
No. 360	2.10	0.06	0.65	0.22	3.00	3.56	4.26
No. 366	1.70	0.09	0.47	0.28	2.83	3.44	4.01
No. 374	1.58	0.08	0.30	0.77	2.79	3.51	4.04

TEST DATA

The chemical analyses of the sets are shown in Table I. Total carbon and silicon have been very closely checked but the other results are of routine accuracy only. Graphite was determined only on the 2 by 1-in. bars (P) using drillings from a $\frac{7}{8}$-in. drill run through the middle of the bar lying flatwise.

The ultimate load and deflection (both corrected), with the calculated values for modulus of rupture, *R*, and modulus of elasticity, *E*, are shown for each bar in Table II. Bars containing flaws have been marked with an asterisk and excluded from the averages. One very strong bar (5400 lb.) in No. 358 was excluded. The averages of Table II are shown in Table III, which includes the factors for both load and deflection.

The results are also shown graphically in Figs. 1, 2 and 3. Fig. 1 shows the average bending curve for each group of each set. Fig. 2 shows the values for modulus of rupture and modulus of elasticity plotted in the order of the strength. The reciprocal of the carbon plus $\frac{1}{3}$ silicon has also been included in Fig. 2 as an interesting comparison. It will be noted that the phosphorus of set No. 374 has caused it to be misplaced in the graph for modulus of elasticity. Fig. 3 shows the breaking loads of the arbitration (A) and the pipe

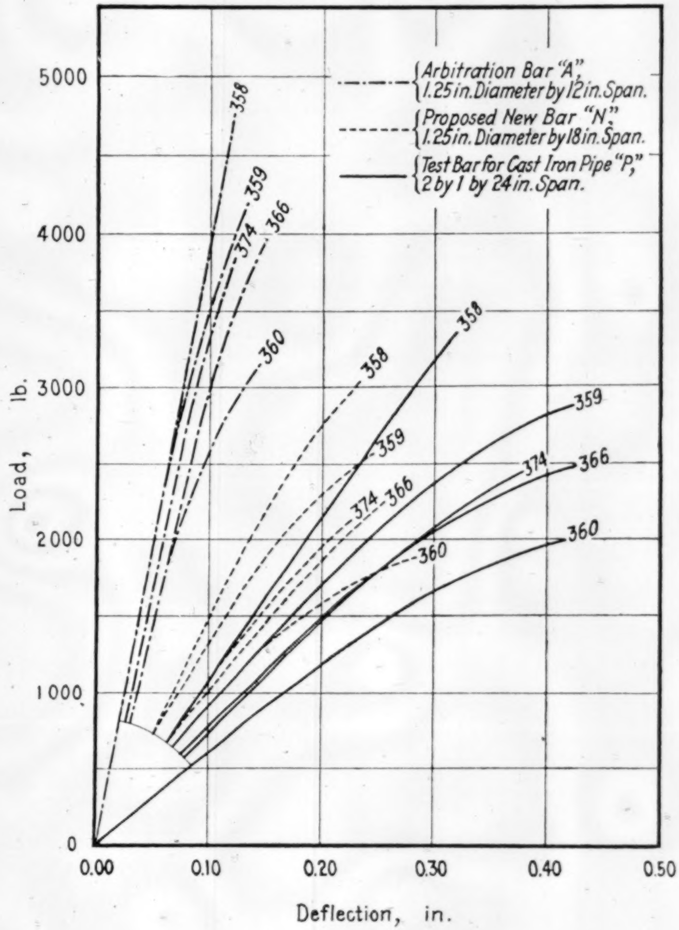

FIG. 1.—Average Load-Deflection Curves for Each Bar and Each Set.

TABLE II.—RESULTS OF TESTS.

Values of Modulus of Rupture and Modulus of Elasticity given in the table have been computed from the common beam formulas. See text.

Bar	Load, *W*, lb.	Deflection, *D*, in.	Modulus of Rupture, *R*, lb. per sq. in.	Modulus of Elasticity, *E*, lb. per sq. in.
SET No. 358				
A—1.25 by 12 in.	4590	0.12	71 800	11 500 000
	4520	0.105	70 700	12 900 000
	4750	0.125	74 300	11 400 000
	4830	0.14	75 600	10 400 000
	*4380	0.085	68 600	15 500 000
	5400	0.13	84 500	12 500 000
Average	4670	0.12	73 100	11 500 000
N—1.20 by 18 in.	3075	0.235	81 600	15 600 000
	*3090	0.20	82 100	18 400 000
	3000	0.22	79 700	16 300 000
Average	3040	0.23	80 600	16 000 000
P—2 by 1 by 24 in.	3320	0.31	59 800	18 500 000
	3420	0.32	61 600	18 500 000
	*2930	0.28	52 700	18 100 000
Average	3370	0.315	60 700	18 500 000
SET No. 359				
A—1.25 by 12 in.	4240	0.13	66 400	9 800 000
	*3600	0.10	56 300	10 800 000
	4120	0.13	64 500	9 500 000
	4250	0.13	66 500	9 800 000
	4200	0.135	65 700	9 400 000
	4150	0.135	64 900	9 200 000
Average	4190	0.13	65 600	9 500 000
N—1.20 by 18 in.	2590	0.24	68 800	12 900 000
	2500	0.235	66 400	12 700 000
	2550	0.23	67 700	13 200 000
Average	2550	0.235	67 600	12 900 000
P—2 by 1 by 24 in.	3000	0.44	54 000	11 800 000
	2890	0.41	52 000	12 200 000
	2880	0.41	51 800	12 100 000
Average	2920	0.42	52 600	12 000 000
SET No. 360				
A—1.25 by 12 in.	2980	0.13	46 600	6 900 000
	3190	0.14	49 900	6 900 000
	*2760	0.105	43 200	7 900 000
	3190	0.15	49 900	6 400 000
	3170	0.13	49 600	7 300 000
	3300	0.15	51 600	6 600 000
Average	3170	0.14	49 500	6 800 000
N—1.20 by 18 in.	1930	0.29	51 200	8 000 000
	*1850	0.25	49 100	8 800 000
	1870	0.27	49 700	8 300 000
Average	1900	0.28	50 000	8 200 000
P—2 by 1 by 24 in.	1940	0.41	34 900	8 200 000
	2070	0.45	37 300	8 200 000
	1970	0.41	35 500	8 300 000
Average	2000	0.42	35 900	8 200 000

* **Flawed Bars.**

TABLE II.—RESULTS OF TESTS (*Continued*).

Bar	Load, *W*, lb.	Deflection, *D*, in.	Modulus of Rupture, *R*, lb. per sq. in.	Modulus of Elasticity, *E*, lb. per sq. in.
SET No. 366				
A—1.25 by 12 in	4340	0.15	68 000	8 700 000
	3810	0.14	59 600	8 200 000
	3920	0.145	61 300	8 100 000
	3720	0.14	58 200	8 000 000
	*3460	0.12	54 100	8 700 000
Average	3950	0.145	61 200	8 300 000
N—1.20 by 18 in	2140	0.24	56 800	10 600 000
	2300	0.26	61 100	10 600 000
	*2025	0.205	53 800	11 800 000
Average	2220	0.25	59 000	10 600 000
P—2 by 1 by 24 in	2490	0.42	44 800	10 300 000
	2510	0.43	45 200	10 100 000
	2470	0.40	44 500	10 600 000
Average	2490	0.42	44 800	10 300 000
SET No. 374				
A—1.25 by 12 in	3720	0.111	58 200	10 100 000
	3800	0.113	59 400	10 100 000
	3760	0.118	58 800	9 600 000
Average	3760	0.114	58 800	9 900 000
N—1.20 by 18 in	*1960	0.20	52 000	11 700 000
	2150	0.21	57 100	12 200 000
	2060	0.215	54 700	11 400 000
	2120	0.23	56 300	11 000 000
Average	2110	0.22	54 700	11 500 000
P—2 by 1 by 24 in	*2310	0.36	41 600	11 200 000
	2420	0.37	43 600	11 400 000
	2460	0.38	44 300	11 300 000
Average	2440	0.375	44 000	11 300 000

* Flawed Bars.

(P) bars plotted against those of the new bars (N). The calculated values are drawn in to show the discrepancy of the pipe-bar values and the agreement of the two round bars. Curiously the agreement is reversed on the values for modulus of elasticity, Fig. 2, where the long bars agree fairly closely and the short bars give values far below these two.

By the conventional beam formulas,

$$R_P = 18\ W,\ R_N = 26.5\ W, \text{ and } R_A = 15.65\ W$$

$$E_P = 1728\ \frac{W}{D},\ E_N = 1194\ \frac{W}{D}, \text{ and } E_A = 300.4\ \frac{W}{D}$$

where W = load in pounds and D = deflection in inches.

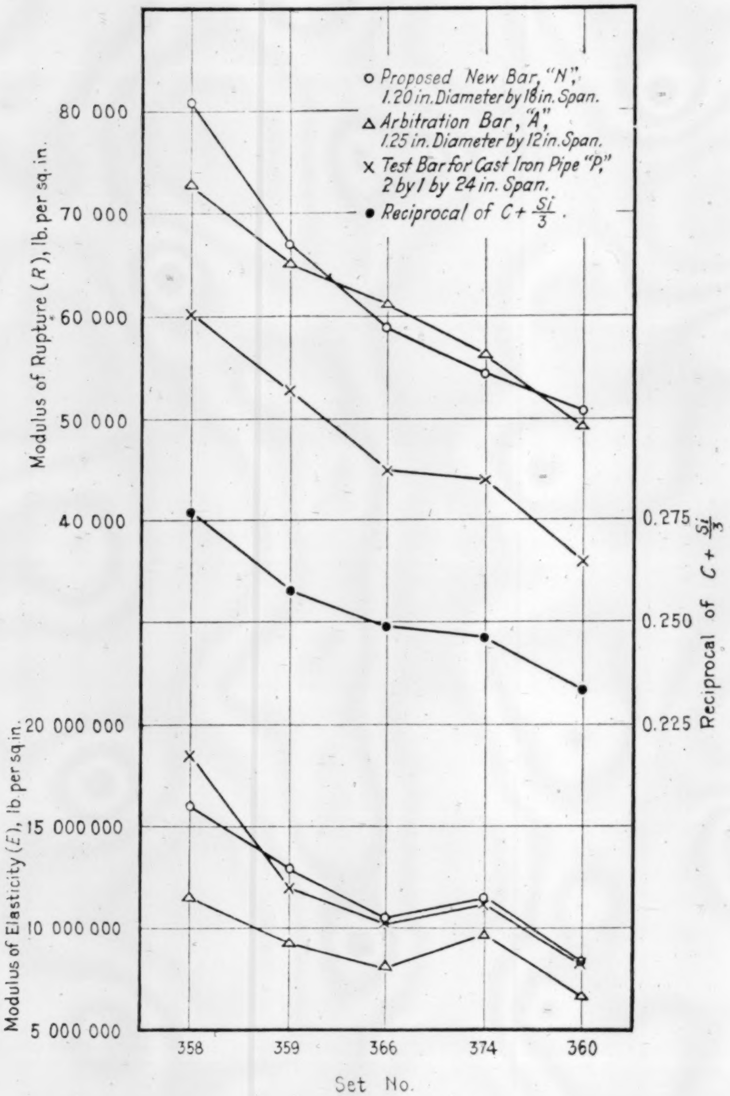

FIG. 2.—Calculated Values of Modulus of Rupture and Modulus of Elasticity. Sets Arranged in Order of Strength.

Making the necessary substitutions and solving we obtain the following theoretical relations:

$$\frac{W_A}{W_N} = 1.69 \text{ and } \frac{W_P}{W_N} = 1.47$$

$$\frac{D_A}{D_N} = 0.425 \text{ and } \frac{D_P}{D_N} = 2.13$$

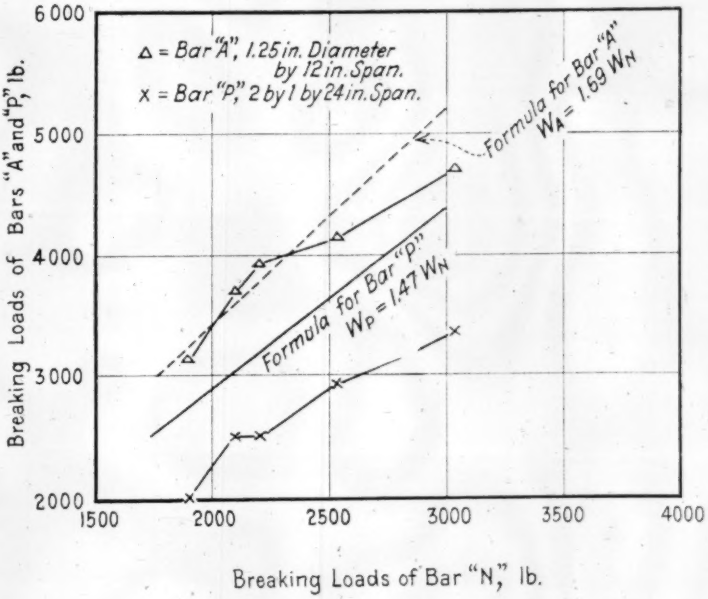

Fig. 3.—Breaking Loads of Arbitration Bars (A) and Pipe Bars (P) Plotted Against Breaking Loads of Proposed New Bar (N). Average of All Sets.

Table III.—Summary of Data, with Load and Deflection Factors.

Set	New Bar, "N," 1.20 by 18 in.		Arbitration Bar, "A," 1.25 by 12 in.				Cast Iron Pipe Bar, "P," 2 by 1 by 24 in.			
	Load, W_N, lb.	Deflection, D_N, in.	Load, W_A, lb.	Load Factor, W_A/W_N	Deflection, D_A, in.	Deflection Factor, D_A/D_N	Load, W_P, lb.	Load Factor, W_P/W_N	Deflection, D_P, in.	Deflection Factor, D_P/D_N
No. 358	3040	0.23	4670	1.53	0.12	0.52	3370	1.12	0.315	1.37
No. 359	2550	0.235	4190	1.64	0.13	0.55	2920	1.14	0.42	1.79
No. 360	1900	0.28	3170	1.66	0.14	0.50	2000	1.05	0.42	1.50
No. 366	2220	0.25	3950	1.78	0.145	0.58	2490	1.12	0.42	1.68
No. 374	2110	0.22	3760	1.78	0.114	0.51	2440	1.16	0.375	1.78
Average	Factor..	....	....	1.68	....	0.53	....	1.12	....	1.62

The average load factors actually found, from Table III, are $\frac{W_A}{W_N} = 1.68$ and $\frac{W_P}{W_N} = 1.12$. Using these empirical values and substituting in the conventional formulas, $\frac{D_A}{D_N} = 0.42$ and $\frac{D_P}{D_N} = 1.62$.

This shows that the curves for the bars N and P (the long bars) are of the same character while those for bars N and A (the round bars) are not. The graph in Fig. 2 shows a very distinctly lower value for E on the arbitration bar than on the other two and it will be noted that the deflection should be only 42 per cent of that of the proposed bar but is actually 53 per cent. In other words the deflection on the rectangular bar is exactly that required by the beam formulas for the actual load relationship, while that of the arbitration bar is 25 per cent greater than that required by the actual or the theoretical load relationship which are practically identical for this bar. This would lead one to suspect that the rectangular bar does not develop the strength of the round bars and that the short span of the arbitration bar gives a higher deflection than is obtained in the longer bars. Probably, the yield due to shear is in some degree responsible.

CONCLUSION

The data obtained lead to the following conversion factors: to convert the new bar to the pipe bar, multiply the load by 1.12 and the deflection by 1.62. To convert the new bar to the arbitration bar multiply the load by 1.68 and the deflection by 0.53.

A STUDY OF THE RELATION BETWEEN PROPERTIES OF CAST-IRON PIPE TESTED UNDER IMPACT, INTERNAL PRESSURE, AND FLEXURE, AND THE CORRESPONDING PROPERTIES FOUND IN SEVERAL KINDS OF TEST SPECIMENS TAKEN THEREFROM

By Arthur N. Talbot[1] and Frank E. Richart[2]

Synopsis

Tests were conducted on twenty-five lots of 6-in. cast-iron pipe made by two sand-cast processes and two centrifugal processes. The greater number of the lots were obtained by purchase. Pipe from ten foundries were tested. In the internal pressure test, hydraulic pressure was applied to the pipe until failure occurred. In the flexure test the load was applied at the middle of a span of 10 ft. In the impact test the pipe, filled with water under pressure, was supported at points 10 ft. apart and a hammer weighing 50 lb. was dropped from increasing heights until failure occurred.

The following auxiliary test specimens were cut from pipe: rings, two forms of tension specimen, and strips for tests in cross-bending. The test strip was cut lengthwise of the pipe and made 0.5 in. in one lateral dimension, the other being the thickness of the pipe as found. It was tested as a beam of 10-in. span loaded at the one-third points and with the 0.5 in. dimension always vertical. For several lots of pipe standard test bars were poured at the time a lot of pipe was cast.

Comparisons are made of the strengths of the various test specimens with the bursting strengths of the pipe and with their flexural strengths. It is found that the strength of the test strip is an index of the strength properties of the pipe. A study was also made of the modulus of elasticity found in the test strip and that in the pipe itself. The tests as a whole give useful data on the relation between the properties and quality of the pipe and the results obtained from test specimens.

This paper reports results of tests by flexure, impact and internal pressure made on 6-in. cast-iron pipe, and compares the properties thus found with the properties found in several forms of test specimens taken from the same lots of pipe. The information was obtained in an investigation made for a group of manufacturers of cast-iron pipe to learn the properties and characteristics of products manufactured by four processes or methods. In all more than 300 pipes were tested.

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The following processes were represented by lots of pipe: (1) the common process of casting vertically in dry sand mold and core, the lots included in Groups I and V; (2) casting horizontally in green sand mold and core, the lots in Group IV; (3) casting by a centrifugal process in refractory mold, the lots in Groups II and VI; and (4) casting by a centrifugal process in water-cooled metal mold, the lots in Group III. The numbers given to the lots of pipe in each group are recorded in Table I, as well as the manufacturers of the pipe. The sand-cast process is the common one and does not need description. The pipe of Group IV were cast horizontally with green sand mold and core, pouring gates being spaced about 6 in. apart along one side of the pipe. In the centrifugal process used in the manufacture of the pipe of Groups II and VI, a metal flask containing an open green-sand mold (except for the head core) is slowly rotated in a

TABLE I.—DATA OF CAST IRON PIPE TESTED.

Group	Process of Manufacture	Description of Process	Lot	Foundry	Source
I	Sand-cast	Pipe cast vertically in dry sand mold and core	1	American Cast Iron Pipe Co	From Manufacturer
			4	American Cast Iron Pipe Co.	From Manufacturer
			20	Lynchburg Foundry Co	From Manufacturer
			30	Warren Foundry and Pipe Co.	From Manufacturer
			40	J. B. Clow & Sons	From Manufacturer
			70	National Cast Iron Pipe Co.	From Dealer
			71	J. B. Clow & Sons	From Chicago
			72	United States Cast Iron Pipe and Foundry Co., Addyston	From Chicago
			73	United States Cast Iron Pipe and Foundry Co., Bessemer	From Detroit
II	Centrifugal	Centrifugal process in refractory (sand) mold	A	American Cast Iron Pipe Co	From Manufacturer
			B	American Cast Iron Pipe Co.	From Manufacturer
			F	American Cast Iron Pipe Co	From Manufacturer
			75	American Cast Iron Pipe Co.	I. P. & L. C.
III	Centrifugal	Centrifugal process in water-cooled metal mold	50	United States Cast Iron Pipe and Foundry Co	From Manufacturer
			51	United States Cast Iron Pipe and Foundry Co	From Manufacturer
			52	United States Cast Iron Pipe and Foundry Co	From Manufacturer
			74	United States Cast Iron Pipe and Foundry Co	From Detroit
IV	Sand-cast	Pipe cast horizontally in green sand mold and core	60	McWane Cast Iron Pipe Co.	From Manufacturer
			76	McWans Cast Iron Pipe Co.	From Dealer
V	Sand-cast	Pipe cast vertically in dry sand mold and core	2	American Cast Iron Pipe Co	From Manufacturer
			3	American Cast Iron Pipe Co	From Manufacturer
			5	American Cast Iron Pipe Co.	From Manufacturer
VI	Centrifugal	Centrifugal process in refractory (sand) mold	C	American Cast Iron Pipe Co.	From Manufacturer
			D	American Cast Iron Pipe Co	From Manufacturer
			E	American Cast Iron Pipe Co.	From Manufacturer

position slightly inclined from the horizontal and the molten metal is poured in at the upper end. During the charging the machine is brought to a horizontal position, and after completion of the charging is made to rotate very rapidly, continuing until the metal has solidified. In the centrifugal process used in the manufacture of the pipe of Group III a metal mold is used (without core except for the head core), which is water-cooled. The mold, in a position slightly inclined to the horizontal, is rotated rapidly and the molten metal is poured into the mold through a cantilever runner which delivers the metal first at the far end, the mold being withdrawn from the runner at a uniform rate. The rotation of the machine is continued until the metal has solidified. An annealing treatment is finally given.

Certain of the lots of pipe, while made in the usual way and intended to represent ordinarily good practice, were furnished by the makers from heats for which careful records of the mixtures, temperatures, etc., had been kept. These included lots Nos. 1 and 4 of Group I from the foundry of one company, and lots Nos. 20, 30 and 40 of Group I representing foundries of three other companies, and lots A, B and F of Group II from another foundry of the first company. Lots Nos. 2 and 3 of Group V were made of special irons and lot No. 5 of 16-ft. lengths of regular iron in the foundry supplying lots Nos. 1 and 4, and lots C, D and E of special irons in the foundry supplying lots A, B and F. Lots Nos. 50, 51 and 52 were bought direct from the manufacturer with the understanding that tests were to be made on them, as was lot No. 60, and may be taken to represent ordinarily good practice in those shops. Lots Nos. 73 and 74, representing two foundries, were bought from the stock of the City of Detroit, the selection being made by the Department of Water Supply. Lots Nos. 71 and 72 were bought from the stock of the Department of Public Works of Chicago and lot No. 70 from the stock of a Chicago dealer, three foundries being thus represented; the selection in these cases was made by Mr. Richart. Lot No. 75 was bought from the Illinois Power and Light Corporation and lot No. 76 from a local supply in Birmingham, Ala. It is felt that the method of selection of the lots bought in the market was such that the pipe so secured may be considered to represent in a fair way what may be expected from a random selection from stocks of excellent quality. The random lots of pipe were obtained and tested after the tests on the other lots of pipe had been completed.

The pipe were all in lengths of 12 ft., except lot No. 5 which was 16 ft. At least twelve pipes of each lot were obtained. Generally, three pipes were subjected to internal pressure, three to cross-bending, and three to impact. For several lots the number tested in the internal pressure test was greater than three. The remaining lengths were retained for use in case it developed that further tests were needed. The pipes were tested at random, no single lot being tested seriatum.

The following tests of the pipes were made: internal pressure tests, flexure tests, and impact tests. The following test specimens were cut from pipe: rings, two forms of tension specimen, and strips for tests in flexure. For several lots of pipe, standard test bars poured at the time a lot of pipe was cast were available.

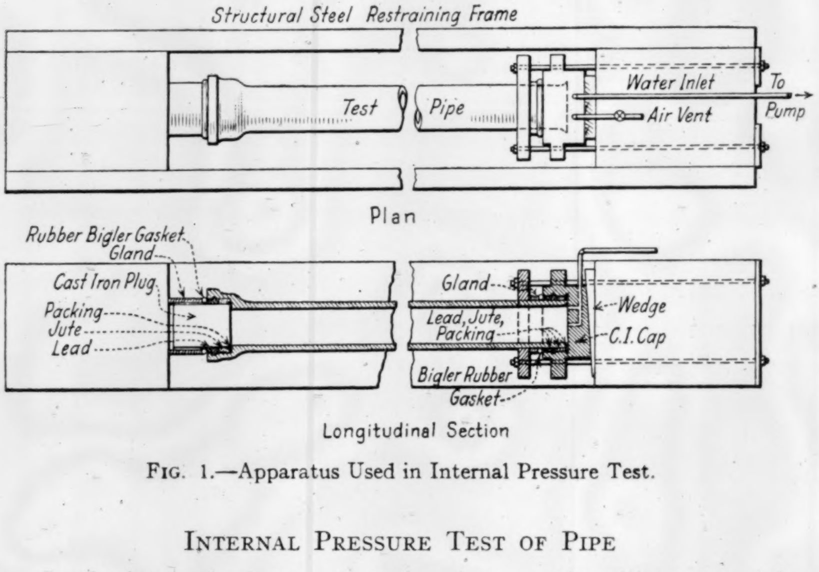

Structural Steel Restraining Frame
Test
Pipe
Water Inlet
Air Vent
To Pump
Plan
Rubber Bigler Gasket
Gland
Cast Iron Plug
Packing
Jute
Lead
Gland
Lead, Jute, Packing
Wedge
C.I. Cap
Bigler Rubber Gasket
Longitudinal Section

FIG. 1.—Apparatus Used in Internal Pressure Test.

INTERNAL PRESSURE TEST OF PIPE

In the internal pressure test, the pipe was placed in a restraining frame, filled with water, and then subjected to internal hydraulic pressure, which was gradually increased until failure occurred by the fracture of the pipe. In preparation for the test, a special cast-iron plug was calked into the bell end of the pipe and a special cast-iron cap was similarly attached to the spigot end. Great care was required in forming the lead joints between pipe and cap or plug to withstand the high pressures in the test. In making the joint, two layers of hemp packing and two layers of soap-treated jute packing were inserted and lead was run into the joint and calked exceptionally hard.

The pipe was then placed in a heavy structural-steel holding or restraining frame (see Fig. 1) built by the American Cast Iron Pipe Co. for the purposes of the test. This frame took the end thrust from the pressure on the plug and cap, a force that may have amounted to 100,000 lb. in some cases in these tests. This thrust caused tension in the sides of the restraining frame, which consisted of built-up plate-and-angle sections so designed as to develop very low working stresses when the pipe was tested. The two ends of the frame, which received the thrust from the cap and the plug, consisted of very stiff diaphragms formed of I-beams and plates. The arrangement of the pipe in the restraining frame was such that as the internal pressure was applied a sufficient movement of the plug was provided for to allow the pressure on plug and cap to be transmitted directly to the stiff restraining frame. The lead joints, however, were held tightly in their initial positions in the cap and in the bell of the pipe by means of cast-iron packing rings or glands bearing on the exposed face of the joint. As it was found that the machined cast-iron plug would move slightly within the lead joint of the bell at quite low pressures and without causing leakage, it is evident that a pipe tested in this apparatus was practically free from longitudinal tension and was also fairly free to shorten in length as it stretched circumferentially under the internal pressure. The existence of this condition was verified by measurements of strain on 2-in. gage lines in a circumferential direction and on 8-in. gage lines in a longitudinal direction that were made on a number of the pipes tested.

In testing, the pipe was filled with water from a main carrying a pressure of about 400 lb. per sq. in. admitted through an opening at the top of the cap, another opening in the cap permitting the discharge of air. Higher pressures were applied by means of a motor-driven triplex pump. Pressures were read by the use of a hydraulic gage placed in the line between pump and test pipe, and compared at frequent intervals with a second gage that had been carefully calibrated with a Crosby fluid pressure scale. The gages indicated that the pressures increased quite steadily and that there was very little surging effect produced by the strokes of the pump. Since any leakage was very troublesome due to the size of the feed line from the pump, great care was taken to secure tight joints. Generally, when a pressure of 1000 lb. per sq. in. was reached, pumping was stopped and the packing glands were again tightened against the lead joints. With these precautions there was little or no leakage. In the case of three or four pipes, leakage was troublesome and the joints were melted out and remade.

Failure was always sudden. It is worth noting that the fracture of every pipe occurred at the part of the circumference and the portion of the length that had the least thickness of wall. The pipe generally failed by the formation of a longitudinal crack, which opened only slightly. Of Groups I and V, in about half the tests a single longitudinal crack formed extending from 3 to 5 ft. in length, sometimes branching at its ends, but no piece being broken from the wall. In the other half of these pipes one or two pieces 9 to 15 in. long and 3 to 5 in. wide, broke out at one end of the crack, and in two cases at both ends of the crack. The cracks were longer in the pipes of Groups II and VI, extending about 5 to 7 ft. in half of them and nearly the full length of pipe in the others. In four of these a single small piece was also broken out of the pipe. The pipes of Group III gave an entirely different fracture. Lots Nos. 51, 52 and 74 broke up into from five to thirty pieces, irregular in form, and generally jagged, nearly always having a spiral form of fracture, the lines of the spiral being at angles that would require 4 or 5 ft. to extend around the pipe once. Lot

TABLE II.—INTERNAL PRESSURE TEST OF PIPE.

Group	Lot	Number of Pipes	Thickness at Break, in.	Average Bursting Pressure, lb. per sq. in.	Average Bursting Strength, lb. per sq. in.	Mean Deviation, per cent of average strength for each lot
I	1	3	0.47	2 160	13 900	4.8
	4	3	0.44	2 340	16 100	10.3
	20	3	0.45	2 160	14 700	9.2
	30	3	0.42	1 900	13 700	2.6
	40	3	0.43	1 890	13 400	15.6
	70	3	0.50	2 430	14 800	3.9
	71	3	0.44	2 100	14 600	9.3
	72	3	0.45	2 120	14 400	3.7
	73	3	0.44	2 370	16 400	7.3
	Average				14 700	7.4
II	A	3	0.43	2 940	20 200	1.9
	B	3	0.36	2 410	20 800	6.5
	F	3	0.42	2 990	19 600	3.5
	75	5	0.42	2 920	20 600	5.7
	Average				20 300	4.4
III	50	6	0.31	1 530	15 700	7.5
	51	6	0.36	2 860	24 400	12.3
	52	6	0.39	3 230	25 800	5.6
	74	4	0.39	3 790	30 000	3.8
	Average				24 000	7.3
IV	60	6	0.35	1 750	15 600	19.3
	76	5	0.34	2 350	20 900	10.6
	Average				18 200	15.0
V	2	3	0.44	2 220	14 400	8.3
	3	3	0.41	2 060	15 200	7.2
	5	3	0.45	1 870	12 500	9.4
	Average				14 000	8.3
VI	C	3	0.42	3 130	22 100	5.1
	D	3	0.40	2 730	20 500	7.9
	E	3	0.33	2 670	24 900	2.9
	Average				22 500	5.3

No. 50, which developed a considerably lower tensile resistance, did not shatter so badly.

After the test the pipe was broken up with a sledge and measurements of thickness were taken at each end of horizontal and vertical diameters for five sections along the length of the pipe. Measurements of the thickness were also made at several points along the line of fracture, and the general thickness so found was recorded as the "thickness at break."

Table II gives the results of the internal pressure tests. The bursting strength or tensile resistance against internal pressure reported in the table was calculated from the internal pressure at failure, the inside diameter of the pipe, and the "thickness at break," using the formula $S = \frac{pd}{2t}$, where S is the tensile resistance in pounds per square inch, p the internal pressure in pounds per square inch, d the inside diameter of pipe in inches and t the "thickness at break" in inches. Only the average bursting strength for each lot is given, but it is seen from the values of the mean deviation of the bursting strengths from the average strength that the several pipes of a lot did not vary greatly from the average of the lot. It will be seen that the bursting strength of one group differed from that of another.

One pipe of lot No. 71 which proved to have a thin spot for 18 in. of its length near the bell end (more than 25 per cent thinner than the average thickness) gave a bursting strength of 10,500 lb. per sq. in., and was omitted from the table. Two pipes of lot No. 50, with no apparent flaws, gave bursting pressures of 570 and 670 lb. per sq. in. and bursting strengths of 5400 and 7000 lb. per sq. in., and also were omitted. One pipe of lot No. 2 proved to have a thin spot and one of lot D a thin section; the use of the dimension of the thin portion gave bursting strength so abnormally high that these pipes also were omitted.

FLEXURE TEST OF PIPE

The flexure or cross-bending test was made by applying load on the pipe at the middle of a span of 10 ft., the supports being approximately symmetrical with respect to the full length of the pipe. Rocker supports were used, the rocker having a radius of 10 in. and the knife edges, $1\frac{1}{4}$ in. The load was applied through a similar knife edge. The deflection of the pipe during the test was measured with a precision of 0.01 in. For every load, the external vertical and horizontal diameters of the pipe were also measured at a section about 3 in. from the mid-span. Generally the measurements were taken at each 1000 lb. of load up to and including the one next to the load of failure. The load was applied at a nominal rate of 0.22 in. per minute.

The pipe always failed very suddenly without previous cracking noises or other warning. Generaly speaking, it was found that when a load gave a greatly increased rate of deflection, failure occurred within another 1000 lb. Generally the pipe broke straight across, the fracture being clean-cut and almost square and otherwise generally having the lines of the fracture all within 2 in. of the mean section of the break. A few broke with a fracture somewhat more oblique. The fracture generally occurred at one side of the load point, generally within 5 to 8 in. of it. In five cases of Group III there were two fractures, one on each side of the load point, a piece of the pipe 15 to 22 in. long thus being broken out. In several cases in this group a longitudinal crack a few inches long was formed through the top

TABLE III.—FLEXURE TEST OF PIPE.

Group	Lot	Number of Pipes	Thickness at Break, in.	Average Maximum Load, lb.	Average Modulus of Rupture, lb. per sq. in.	Mean Deviation, per cent of average strength for each lot
I	1	3	0.53	15 920	29 000	3.8
	4	3	0.51	16 830	32 200	6.4
	20	3	0.50	13 920	27 000	2.2
	30	3	0.47	13 220	26 800	0.7
	40	3	0.50	15 970	29 300	7.1
	70	3	0.53	15 970	28 400	5.3
	71	3	0.50	14 030	26 300	2.7
	72	3	0.52	14 730	26 900	6.5
	73	3	0.55	15 530	26 800	4.9
	Average				28 100	4.4
II	A	3	0.46	17 850	39 500	2.4
	B	3	0.42	12 730	30 700	3.5
	F	3	0.46	17 340	37 500	4.4
	75	3	0.46	17 700	38 600	8.5
	Average				36 600	4.7
III	50	6	0.35	13 130	34 500	5.8
	51	6	0.37	12 660	32 200	6.5
	52	6	0.39	15 450	37 000	12.2
	74	3	0.41	18 230	42 700	4.3
	Average				36 600	7.2
IV	60	6	0.40	13 570	33 100	8.1
	76	5	0.39	14 960	36 100	4.7
	Average				34 600	6.4
V	2	3	0.50	15 430	29 900	0.3
	3	3	0.47	18 090	36 100	1.7
	5	2	0.51	17 170	31 700	1.6
	Average				32 600	1.2
VI	C	3	0.48	18 980	38 700	3.8
	D	3	0.48	17 580	36 800	4.9
	E	3	0.48	20 390	41 800	2.0
	Average				39 100	3.6

wall of the pipe at and near the load point a little time before failure occurred.

The modulus of rupture was calculated from the formula $S = \frac{Plc}{4I}$ where S is the modulus of rupture in pounds per square inch, P the breaking load in pounds, I the moment of inertia of the section in inches^4, l is the span length in inches and c the distance from the neutral axis to the remotest fiber in tension in inches. For this purpose the moment of inertia of a section was calculated from the properties of two circular areas, an outside and an inside circle which gave the measured thickness of wall at the top and bottom of the pipe at the section of fracture. The neutral axis was determined in a similar way. The distance from the neutral axis to the extreme fiber in tension so found was used for the c of the formula. This method, of course, is approximate and empirical, but as the variations in the thickness of the pipe at the different parts of the section were relatively small and the pipe was placed in the testing machine just as it happened to come, this method of calculation may be expected to give results that are properly comparative.

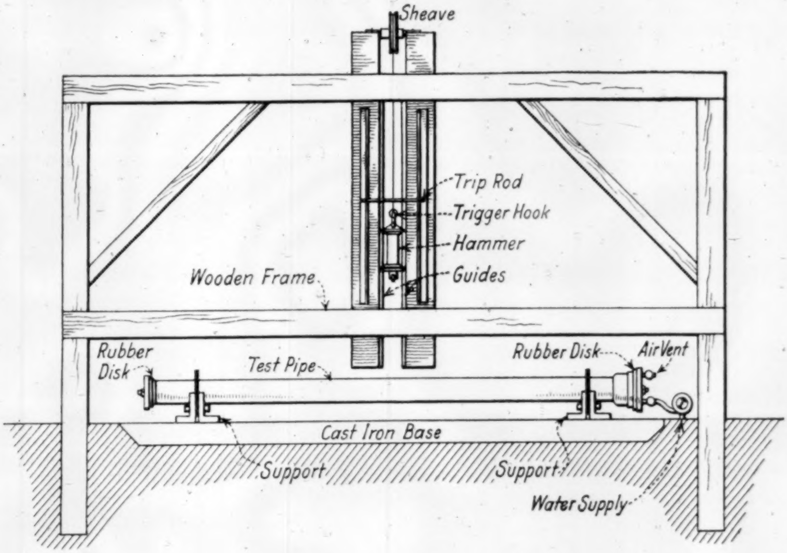

Sheave
Trip Rod
Trigger Hook
Hammer
Guides
Wooden Frame
Rubber Disk
Test Pipe
Rubber Disk
Air Vent
Cast Iron Base
Support
Support
Water Supply

FIG. 2.—Impact Testing Machine.

Table III gives the results of the flexure tests.

IMPACT TEST OF PIPE

The impact tests were made with the impact pipe testing machine built by the American Cast Iron Pipe Co. The pipe was placed on two supports, filled with water, kept under the water pressure of the distribution main during the test (generally about 65 lb. per sq. in.), and subjected to blows of the hammer dropped from increasing heights until a crack became apparent through a leakage of water.

The general arrangement of the impact testing machine is shown diagrammatically in Fig. 2. The pipe rested on two supports 10 ft. apart attached to a cast-iron base 14 ft. long and weighing about 2000 lb., which was embedded in the ground. The supports were recessed to receive the pipe which was held tightly in place by means of a clamp at each support. The 50-lb. hammer, falling between vertical guides, was arranged to strike the pipe at midspan. It carried as its striking edge a hardened steel bar $\frac{1}{2}$ in. wide by $1\frac{1}{2}$ in. deep by 4 in. long, with its lower edge slightly rounded. The low center of gravity of the hammer and the large ratio of vertical to horizontal distance between the contacts with guides served to minimize friction and to produce uniform and consistent behavior of the machine. The uniformity of

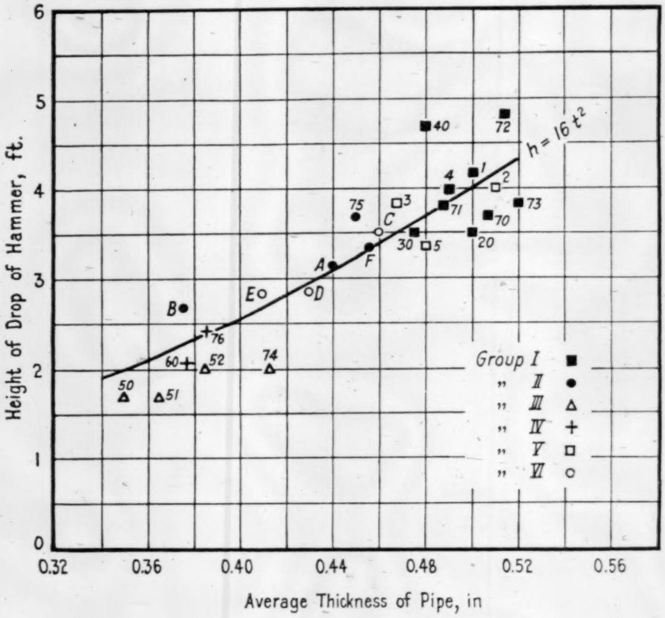


FIG. 3.—Thickness of Wall of Pipe and Height of Drop in Impact Test.

action was studied by subjecting a piece of cold-rolled shafting to blows of the hammer and noting the uniformity of the indentation produced in the shafting. These tests were repeated each day and showed an average variation from the mean indentation of 2 to 3 per cent, which was considered a very satisfactory performance.

Before a pipe was placed in the testing machine, rubber disks were clamped in close contact over the two ends by means of a bolt passing through the length of the pipe, an arrangement that gave a tight connection for the water pressures used.

In making the test, the first blow was made at a height of 6 in. The succeeding applications were made with increments of height of 6 in., the greatest height of drop permitted by the machine being 5 ft. As the hammer was dropped from increasing heights, failure was noted when a leak or spray of water came through the pipe. Generally, an additional blow was applied after the one causing failure. The pipe was finally broken up with a sledge and measurements were taken of the thickness of walls.

In Fig. 3 the average height of drop for each lot of pipe has been plotted against average thickness of wall at the cross-section at point of failure.

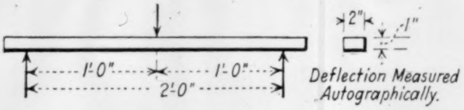
In every case the failure of the pipe became apparent through the formation of a crack that ran longitudinally along the top of the pipe for a length of 1 to 12 in. (in one pipe to 36 in.). In about half of the tests the crack extended on both sides of the point where the hammer struck and in the other half on only one side or the other of this point. It is evident that the failure was due to flattening of the cross-section of the pipe at its top and the breaking of the wall by bending inwardly. The crack would form first at the under side of the top wall of the pipe under this bending, and thus the crack may not have become apparent at the blow at which it started, but was observed when it was carried entirely through the wall and the water spurted through the opening.

After the impact tests here reported had been studied, it was concluded that the impact testing machine should be modified by making the distance between centers of supports 2 ft. and arranging it so as to permit testing a pipe at several points along its length. Tests made on pipe near middle, bell end, and spigot end show that the test in the new form is an improvement over the original. It permits finding whether one part of a pipe is weaker than another and avoids many of the uncertainties of the relation between the energy of the blow, the inertia and deflection of the pipe, and its resistance to impact. The test is more severe, and increments of drop of 3 in. were used. The results are not ready for presentation.

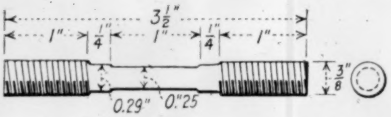
Auxiliary Tests

The auxiliary test specimens, the method of testing, and the test results are given below.

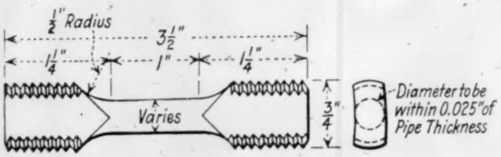
Standard Pipe Test Bar.—For the lots of Group I, II, V and VI not bought in the market, standard pipe test bars 2 by 1 by 26 in., shown in Fig. 4(*a*), were made when the pipes were poured. These conform to the A.S.T.M. Standard Specifications for Cast-Iron Pipe and Special Castings (A 44 – 04).[1] The metal was poured into the top of the vertical dry sand molds. Generally six test bars were made, representing metal used at different times in the pouring of the pipe of one lot, but the number varied from four to nine. The test bars were tested flatwise with load in the middle of a 24-in. span. An autographic load-deflection diagram was obtained. Flaws and other defects that appeared were noted.


1'-0"
1'-0"
2'-0"
2"
1"
Deflection Measured Autographically.

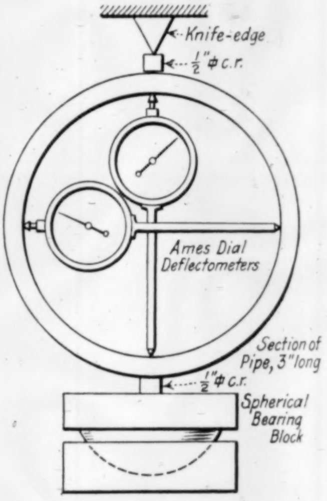
(*a*) Test Bar.


3 1/2"
1"
1/4"
1"
1/4"
1"
3/8"
0.29"
0."25

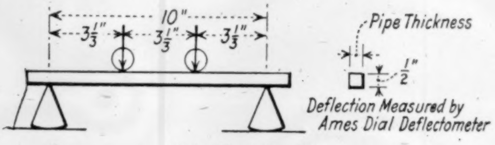
(*b*) Tension Specimen No. 1.


1/2" Radius
3 1/2"
1 1/4"
1"
1 1/4"
Varies
3/4"
Diameter to be within 0.025" of Pipe Thickness

(*c*) Tension Specimen No. 2.


Knife-edge
1/2" φ c.r.
Ames Dial Deflectometers
Section of Pipe, 3" long
1/2" φ c.r.
Spherical Bearing Block

(*d*) Ring.


10"
3 1/3"
3 1/3"
3 1/3"
Pipe Thickness
1/2"
Deflection Measured by Ames Dial Deflectometer

(*e*) Test Strip.

Fig. 4.—Auxiliary Test Specimens.

Results of the tests are given in Table IV. The modulus of rupture was calculated in the usual manner. The loads and the deflections recorded in the table are modified values which correspond to an exact 1 by 2-in. cross-section.

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[1] 1924 Book of A.S.T.M. Standards.

Tension Test Specimen No. 1.—Of the test specimens cut from the walls of the pipe, tension test specimen No. 1, as shown in Fig. 4(*b*), was 3½ in. in extreme length, threaded at the ends, and turned down to a diameter of ¼ in. for a length of about 1 in. The axis of the specimen coincided with the middle of the wall of the pipe. Three specimens were cut from one pipe that had been tested in flexure for each lot of every group except lots Nos. 70 to 76. Some difficulty was encountered in making these test specimens, many being broken in the process, but all the tests seemed trustworthy and concordant.

The tests were made in a testing machine of 10,000-lb. capacity. The holding device was such as to minimize eccentricity of loading. The results are given in Table IV.

Tension Test Specimen No. 2.—For the purpose of getting some representation of the metal at the outer and inner faces of the wall of the pipe, test specimen No. 2 was used, as illustrated in Fig. 4 (*c*). The extreme length is 3½ in. Threaded ends were used. The middle portion was turned down to a diameter slightly less than the thickness of the wall. Three specimens were cut from one pipe that had been tested in flexure for each lot except lots Nos. 70 to 76. The specimens were more readily made than those of No. 1, and the results are concordant.

The same testing machine and a similar holding device were used as in the tests of tension specimen No. 1. The results are given in Table IV.

Ring.—One ring 3 in. long was cut from one pipe that had been tested in flexure for each lot. The load was applied along the top element through a ½-in. square bar, and the support at the bottom element was a similar bar which rested on a spherical bearing block, shown in Fig. 4(*d*). Changes in horizontal and vertical diameters were read throughout the test.

The results are given in Table IV. The modulus of rupture is calculated from the usual formula $S = \frac{Mc}{I}$, in which M, the bending moment at the top, is determined from the formula $M = 0.159\ Pd$, P being the breaking load and d the mean diameter of the pipe.

Test Strip.—In an effort to get a test specimen that would be easily made and would in any part of a section be representative of the metal throughout the thickness of the wall of the pipe, a test strip the full thickness of the pipe and ½ in. in the other lateral dimension and 12 in. long was cut from the pipe. Generally four test strips were cut from one pipe of each lot that had been tested in flexure. For lots Nos. 70 to 76 in addition there were cut one test strip from each pipe used in the flexure test and two test strips from each pipe used in the internal pressure test.

TABLE IV.—RESULTS OF TESTS OF AUXILIARY TEST SPECIMENS.

		Test Bar			Test Strip				Tension Test		Ring
Group	Lot	Maximum Load, lb.	Modulus of Rupture, lb. per sq. in.	Maximum Deflection, in.	Width, in.	Maximum Load, lb.	Modulus of Rupture, lb. per sq. in.	Maximum Deflection, in.	Specimen No. 1, Strength, lb. per sq. in.	Specimen No. 2, Strength, lb. per sq. in.	Modulus of Rupture lb. per sq. in.
I	1	2 070	37 200	0.30	0.50	436	35 300	0.200	23 200	23 500	41 200
	4	2 170	39 100	0.34	0.51	491	38 300	0.226	21 600	25 400	37 500
	20	2 160	38 200	0.35	0.44	371	33 900	0.187	20 300	20 100	41 900
	30	1 970	35 400	0.31	0.48	402	33 700	0.216	20 200	19 800	38 300
	40	2 520	45 400	0.38	0.46	450	39 100	0.224	23 100	26 400	46 700
	70	....	.....	....	0.53	462	35 000	0.212	.....	.....	.....
	70[a]	....	.....	....	0.53	456	34 900	0.204	.....	.....	.....
	71	....	.....	....	0.49	334	29 300	0.181	.....	.....	.....
	71[a]	....	.....	....	0.44	364	32 500	0.208	.....	.....	.....
	72	....	.....	....	0.51	402	32 300	0.212	.....	.....	.....
	72[a]	....	.....	....	0.48	389	32 600	0.207	.....	.....	.....
	73	....	.....	....	0.55	472	35 500	0.209	.....	.....	.....
	73[a]	....	.....	....	0.49	417	34 400	0.198	.....	.....	.....
	Average...		39 100	....	....	...	34 400	.....	21 700	23 000	41 100
II	A	2 415	43 500	0.36	0.44	477	43 500	0.246	25 000	27 700	44 500
	B	2 210	39 500	0.36	0.38	388	41 100	0.220	26 100	27 500	45 800
	F	2 460	44 300	0.39	0.50	530	43 000	0.222	28 400	27 600	50 000
	75	....	.....	....	0.45	518	46 000	0.253	.....	.....	.....
	75[a]	....	.....	....	0.44	458	42 100	0.236	.....	.....	.....
	Average...		42 400	....	....	...	43 100	.....	26 500	27 600	46 800
III	50	....	.....	....	0.36	455	50 800	0.192	37 100	34 000	46 500
	51	....	.....	....	0.38	405	43 500	0.156	33 100	34 800	45 800
	52	....	.....	....	0.38	493	48 800	0.202	33 100	35 600	48 600
	74	....	.....	....	0.40	491	50 700	0.193	.....	.....	.....
	74[a]	....	.....	....	0.40	488	49 400	0.193	.....	.....	.....
	Average...		....	....	.....	......	48 600	.....	34 400	34 800	47 000
IV	60	....	.....	....	0.47	466	40 000	0.232	30 600	34 000	46 000
	76	....	.....	....	0.40	449	43 700	0.241	.....	.....	.....
	76[a]	....	.....	....	0.40	404	40 600	0.234	.....	.....	.....
	Average...		....	....	.....	......	41 400	.....	30 600	34 000	46 000
V	2	2 085	37 500	0.34	0.48	459	39 100	0.259	23 700	23 800	43 800
	3	2 650	47 700	0.38	0.46	481	42 300	0.214	26 700	31 000	48 500
	5	2 230	40 200	0.33	0.43	422	39 400	0.223	22 700	24 500	44 800
	Average...		41 800	....	....	...	40 300	.....	24 400	26 400	45 700
VI	C	2 620	47 200	0.36	0.48	541	45 400	0.213	30 300	30 600	58 500
	D	2 220	39 600	0.33	0.46	461	40 900	0.220	25 700	27 300	50 000
	E	2 650	47 500	0.35	0.46	465	41 100	0.184	33 600	35 800	49 100
	Average...		44 800	....	....	...	42 500	.....	29 900	31 200	52 500

[a] Test strips cut from pipe after internal pressure test.

The test strip was tested as a beam with the ½-in. dimension placed vertically and the load was applied equally at the one-third points of a span of 10 in., as illustrated in Fig. 4(*e*). Deflection at the middle of the span was measured throughout the test by means of a deflectometer reading to 0.001 in. The results of the tests are given in Table IV.

DISCUSSION

In a consideration of the relative value of different tests of pipe, it seems evident that as the purpose of the pipe is to hold the water or other contained material under pressure the resistance of the pipe to pressures (bursting strength) is very important. Granted that the thickness of the pipe is sufficient to sustain external loads that may come upon it, either static or impact loads, and that the metal is durable for the conditions of use, the information given by the internal pressure test of a length of pipe on its tensile strength and the nature of the failure is of high value and usefulness. The flexure test of pipe gives information on the load that may be supported and may give data on the quality of the pipe, especially if flaws or defects develop. An impact test shows resistance to falling loads and to blows received in handling and may bring out properties of the metal in the pipe. The data of the several auxiliary tests may well be studied to learn their value in furnishing information on the quality of the metal in the pipe, as well as to find what relation exists between these tests and the tests of the pipe itself. The tests of the pipe will be considered in the following order: impact test, flexure test, and internal pressure test; and then a general comparison of all the tests will be made.

In the impact test, the pipe rested on supports 10 ft. apart. The energy of the falling hammer, disregarding friction, etc., is proportional to the height of drop. Disregarding the spring of the testing apparatus, this energy is taken principally in two ways: by the work done in deflecting the pipe as a beam and by that in overcoming the inertia of the pipe and the contained water during the movement through this deflection. From theoretical considerations the work against inertia probably is several times that done in bending the pipe. The work done in distorting the pipe section is evidently relatively small. As the supports are well apart, it is plain that the section of the pipe does not deform in the manner that is found when a ring is supported along a bottom element and loaded along the top, but since the load is applied at one point at the top the section immediately under the hammer is flattened somewhat at the top, the resulting bending moment across the top element being much larger than that at the end of the horizontal diameter or than that across the bottom element. How far along the length of the pipe the sections are thus deformed is not known; it appears that this length is dependent on the thickness of the pipe and also upon the value of the modulus of elasticity of the metal. No method of estimating the length giving effective resistance to the blow has been found, and it seems futile to attempt an analytical treatment. In Fig. 3 a curve for the equation $h = 16t^2$ has been drawn, h being the drop in feet and t the average thickness of the pipe in inches. It appears that the resistance to the impact of the test varies quite closely as the square of the thickness of the wall of the pipe. It is seen that there is a noticeable difference in the resistance of various lots in the impact test as shown by the position with respect to the line drawn, which represents fairly well the mean of the results.

The values of the modulus of rupture found in the flexure tests of the pipe in the nine lots in Group I (see Table III) do not differ much from the average of the lots, 28,100 lb. per sq. in. The average for the individual lots in Group II range from 30,700 to 39,500 lb. per sq. in., and in Group III from 32,200 to 42,700 lb. per sq. in. The two lots in Group IV give values of 33,100 and 36,100 lb. per sq. in.

The tensile resistance developed in the internal pressure tests (bursting strength), shown in Table II, runs fairly uniformly for Group I, the averages for the several lots varying little from 14,700 lb. per sq. in. Group II also gives small range in bursting strength, the average values varying little from 20,000 lb. per sq. in. The lots in Group III range from 15,700 to 30,000 lb. per sq. in. and the values in Group IV are 15,600 and 20,900 lb. per sq. in.

Before attempting a comparison and correlation of the various auxiliary test specimens with the properties of the pipe from which they were taken, it may be well to give some characteristics of the test specimens. It is usually accepted that the standard test bar may be expected to represent the quality of the metal that is poured into the mold. It may not be an index of the metal of the pipe itself; that may best be determined by taking test specimens from the pipe. The tension test specimens may determine the tensile properties of the metal in the pipe in the direction of its length. The form of tension specimen No. 1 is such that only the middle part of the thickness of wall is represented. In tension specimen No. 2 the outer portion of the wall is represented, but not fully. In the test strip the full thickness of wall is represented and at any given distance from the neutral axis metal from every part of this thickness is equally represented. The flexure test gives modulus of rupture and not tensile strength, but a relation between the modulus of rupture of the test strip and the tensile resistance to internal pressure (bursting strength) developed in the internal pressure test may be sought for pipe made by a given process.

The test of the ring is also a flexure test. It happens, however, that the empirical formula used for calculating the modulus of rupture of the ring involves the assumption that the modulus of elasticity developed at the horizontal sections of the ring is the same throughout the thickness as that acting at the same time in the vertical sections, and the same uniformity is assumed for all sections between. This assumption markedly affects the relation between the moment developed at the horizontal and the vertical sections and hence of the actual value of the moment at either section, the sum of the two, of course, being $\frac{1}{4}$ *Pd*. For cast iron it is evident that by reason of the varying value of this modulus of elasticity (so-called) the resisting moment developed at the vertical sections will not be 1.75 times that developed at the horizontal sections, as the use of a constant modulus of elasticity in an analysis gives, and that for different irons the relation between these two resisting moments will vary and hence the moduli of rupture calculated in the usual way will not allow accurate comparisons of the quality of the metal. For metal having a sharply curved stress-strain relation the moment developed at the sections of the vertical diameter will probably be considerably less than 0.159 *Pd*. Besides, in the flexure of the ring the portions of the wall next to its surfaces receive high stress and those in the interior low stress, so that the metal is far from being equally stressed at points throughout the wall, whereas in the internal pressure test of pipe every portion of the thickness is stressed nearly equally, as is also the case in the wall at the top and bottom of the pipe in the flexure test. A pipe having metal of high strength near the surfaces of the wall and weaker material in the interior (or having defects in the interior) would thus show different results in the ring test than would another having the conditions reversed even though the average tensile resistance were the same. Because of inherent disadvantages of the ring test, little consideration was given to it, the number of ring specimens tested being generally limited to one from each lot of pipe.

In studying the value of the several forms of test specimens for giving information on the properties of the cast-iron pipe, it will be convenient to use the ratio of the strength of the different test specimens to the strength of the pipe in internal pressure and in flexure. For this purpose the ratios referred to have been calculated from the strengths given in Tables II, III and IV and are reported in Table V. In studying the results of the auxiliary test specimens it should be kept in mind, as has already been stated, that except for the test specimens taken from lots Nos. 70 to 76 all the test strips and all the tension test pieces for a given lot of pipe were cut from one pipe. The original purpose of this was to study the usefulness of the auxiliary test specimens for determining the quality of the metal in a pipe and the uniformity obtainable in specimens cut from the same pipe. For the purpose of judging somewhat of the variation in results in different parts of a pipe a test strip was cut at four different points along the length of one pipe that had been tested in flexure for each of lots Nos. 70 to 76. Besides, out of the other two or more pipes of each lot one test strip was also cut. The mean deviation of the strengths from the average of the four test strips cut from one pipe of a lot ranged from 4 to 9 per cent, and the maximum variation from 5 to 12 per cent. The variation from pipe to pipe for these lots may be judged from the mean deviation given in Table VI. It appears that the variation throughout the length of a pipe was not as great as the

TABLE V.—RATIOS OF THE STRENGTHS FOUND IN THE SEVERAL FORMS OF TEST SPECIMEN TO THE BURSTING STRENGTH AND THE MODULUS OF RUPTURE OF THE PIPE.

For the tension test specimens the tensile resistance is used as the strength, and for the remaining ones the modulus of rupture.

Group	Lot	Bursting Strength of Pipe, lb. per sq. in.	Ratio to Bursting Strength					Modulus of Rupture of Pipe, lb. per sq. in.	Ratio to Modulus of Rupture				
			Test Bar	Test Strip	Tension Test Specimen No. 1	Tension Test Specimen No. 2	Ring		Test Bar	Test Strip	Tension Test Specimen No. 1	Tension Test Specimen No 2	Ring
I	1	13 900	2.67	2.54	1.67	1.69	2.96	29 000	1.28	1.22	0.80	0.81	1.42
	4	16 100	2.43	2.38	1.34	1.58	2.33	32 300	1.21	1.19	0.67	0.79	1.16
	20	14 700	2.60	2.30	1.38	1.37	2.85	27 000	1.41	1.25	0.75	0.74	1.55
	30	13 700	2.58	2.46	1.47	1.45	2.80	26 800	1.32	1.26	0.75	0.74	1.43
	40	13 400	3.39	2.92	1.72	1.97	3.48	29 300	1.55	1.33	0.79	0.90	1.59
	70	14 800	....	2.36	....	....	....	28 400	....	1.23	....	....	....
	71	14 600	....	2.30	....	....	....	26 300	....	1.12	....	....	....
	72	14 400	....	2.26	....	....	....	26 900	....	1.20	....	....	....
	73	16 400	....	2.10	....	....	....	26 800	....	1.32	....	....	....
	Average....		2.73	2.40	1.52	1.61	2.88	.....	1.35	1.24	0.75	0.80	1.43
II	A	20 200	2.15	2.15	1.24	1.37	2.20	39 500	1.10	1.10	0.63	0.70	1.13
	B	20 800	1.90	1.98	1.26	1.32	2.20	30 700	1.29	1.34	0.85	0.89	1.49
	F	19 600	2.26	2.19	1.45	1.41	2.55	37 500	1.18	1.15	0.76	0.74	1.33
	75	20 600	....	2.04	....	....	....	38 600	....	1.19	....	....	....
	Average....		2.10	2.09	1.32	1.37	2.32	.....	1.19	1.19	0.75	0.78	1.32
III	50	15 700	....	3.24	2.36	2.16	2.96	34 500	....	1.47	1.07	0.99	1.35
	51	24 400	....	1.78	1.36	1.43	1.88	32 200	....	1.35	1.03	1.08	1.42
	52	25 800	....	1.89	1.28	1.38	1.88	37 000	....	1.32	0.89	0.96	1.31
	74	30 000	....	1.65	....	....	....	42 700	....	1.19	....	....	....
Average (Omitting No. 50).				1.77	1.32	1.41	1.88	.....	....	....	....	....	....
Average (Including No. 50).				2.14	1.67	1.66	2.23	.....	....	1.33	1.00	1.01	1.36
IV	60	15 600	....	2.56	1.96	2.18	2.95	33 100	....	1.21	0.92	1.03	1.39
	76	20 900	....	1.94	....	....	....	36 100	....	1.21	....	....	....
	Average....		....	2.25	1.96	2.18	2.95	.....	....	1.21	0.92	1.03	1.39

variation between pipes. Altogether, then, it appears that the information given in the tables is applicable to determining with a fair degree of accuracy the general relation between the results of the auxiliary test specimens and the properties found in the pipe.

TABLE VI.—MEAN DEVIATION IN STRENGTHS OF AUXILIARY TEST SPECIMENS FROM THE AVERAGE STRENGTH OF A LOT.

For each type of test specimen, the deviation is recorded as a percentage of the average strength of the test specimens of the lot.

Group	Lot	Test Bar		Test Strip		Tension Test Specimen No. 1		Tension Test Specimen No. 2	
		Number of Tests	Deviation, per cent	Number of Tests	Deviation, per cent	Number of Tests	Deviation, per cent	Number of Tests	Deviation, per cent
I.....	1	6	5.6	4	4.5	2	2.6	3	5.0
	4	4	7.8	4	2.4	3	6.3	3	3.0
	20	6	6.2	4	1.8	2	7.6	3	14.9
	30	6	1.6	4	2.7	1	...	3	3.5
	40	6	5.2	4	5.3	2	8.4	3	3.9
	70	..	...	6	3.0	..	...	..	...
	70[a]	..	...	6	6.8	..	...	..	...
	71	..	...	6	11.8	..	...	..	...
	71[a]	..	...	8	7.8	..	...	..	...
	72	..	...	6	5.3	..	...	..	...
	72[a]	..	...	6	3.9	..	...	..	...
	73	..	...	6	5.3	..	...	..	...
	73[a]	..	...	6	4.8	..	...	..	...
	Average..	....	5.3	..	5.0	..	6.5	..	6.1
II....	A	6	3.2	4	2.8	3	4.3	3	0.5
	B	8	6.5	4	1.6	3	5.0	3	2.8
	F	5	9.4	4	3.4	3	2.1	3	2.5
	75	..	...	6	3.8	..	...	..	...
	75[a]	..	...	12	5.8	..	...	..	...
	Average.	..	6.4	..	3.5	..	3.8	..	1.9
III...	50	..	...	4	2.9	3	1.5	3	8.3
	51	..	...	4	3.9	3	1.7	2	3.7
	52	..	...	4	8.4	3	2.3	3	1.9
	74	..	...	6	6.9	..	...	..	...
	74[a]	..	...	8	6.6	..	...	..	...
	Average.	....	....	....	5.7	..	1.8	..	4.6
IV....	60	..	...	4	3.2	3	5.3	3	3.8
	76	..	...	8	10.2	..	...	..	...
	76[a]	..	...	10	9.2	..	...	..	...
	Average.	....	....	....	7.5	..	5.3	..	3.8
V.....	2	6	2.1	4	9.9	2	2.7	3	3.1
	3	6	3.8	4	2.1	2	6.0	3	5.3
	5	4	4.2	4	2.9	2	0.2	3	2.3
	Average.	....	3.4	..	5.0	..	3.0	..	3.6
VI....	C	9	4.3	4	2.1	3	4.8	3	2.7
	D	8	10.5	4	3.2	3	1.4	3	1.2
	E	7	4.6	4	1.5	3	2.5	3	1.4
	Average..	....	6.5	..	2.3	..	2.9	..	1.8

[a] Test strips cut from pipe after internal pressure test.

From Table V it is seen that ratios are fairly uniform in the different groups but a variation in the averages from group to group is apparent. Naturally the ratio for one form of test specimen differs from that for another.

In making comparisons with the bursting strengths it is seen that the strengths for the test strip and the ring, being flexure tests, are considerably higher than the bursting strength of the pipe, as would be expected since the modulus of rupture of a material like cast iron is considerably higher than its tensile strength. The ratios for the tension tests are also greater than unity; the tensile resistance of the metal in the pipe, at least of that in the samples, which were taken lengthwise of the pipe, was evidently greater than that of the circumferential resistance of the pipe in the internal pressure test. It is probable also that the pressure test searched out and found not only the thinnest portion of the pipe but also the weakest metal. For the several groups, it would seem that the ratio of the strength of the test strip to the bursting strength of the pipe may be taken as 2.4 for Group I, 2.1 for Group II, and 1.8 for Group III (disregarding lot No. 50). For the two tension specimens, the ratios may be given as 1.6 for Group I and 1.35 for Groups II and III. For the standard test bars, made from the poured metal, the ratios average 2.7 for Group I and 2.1 for Group II. It would seem that for ordinary grades of metal the values of the ratios named may be considered representative of the ratios that may be expected in pipes made by the several proceeses under good practice. It will be noted that all the ratios for Group II are 15 to 22 per cent lower than the corresponding ones for Group I.

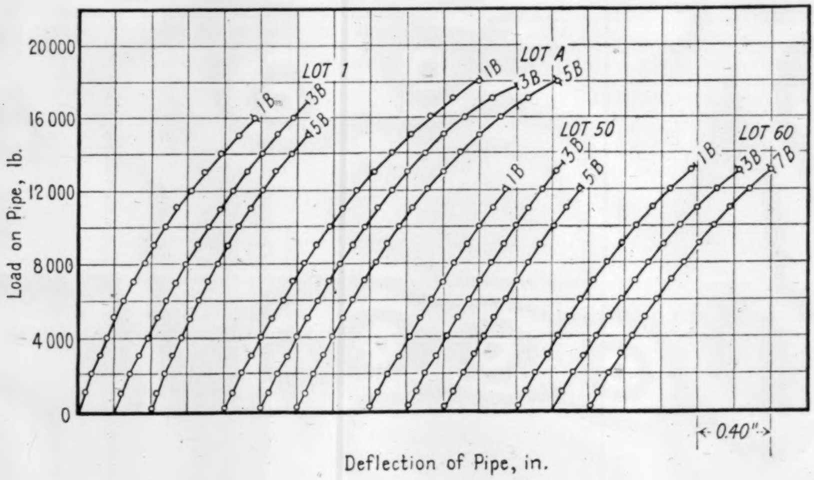

FIG. 5.—Load-Deflection Curves for Tests of Pipe.

In making comparisons with the flexural strengths of the pipe it is seen that the ratios of the strength of the test specimens to the modulus of rupture of the pipe are greater than unity for the test strip and the ring and less for the tension test specimens, except in Group III where the latter ratios reach unity. The values for the test strips vary little in any group. The values of the ratio for the test strips average about 1.25 for Group I, 1.20 for Group II, and 1.35 for Group III, which may be taken as representative values.

In two lots of pipe, the ratios show considerable variation from other lots in the same group. Lot No. 40 in Group I gives markedly higher values for all the ratios than the others of the group. It is interesting to note that in the impact test the pipes of this lot gave the highest resistance obtained. Lot No. 50 in Group III shows markedly higher ratios in the internal pressure test than the others in the group; this is probably connected with the low bursting strength found.

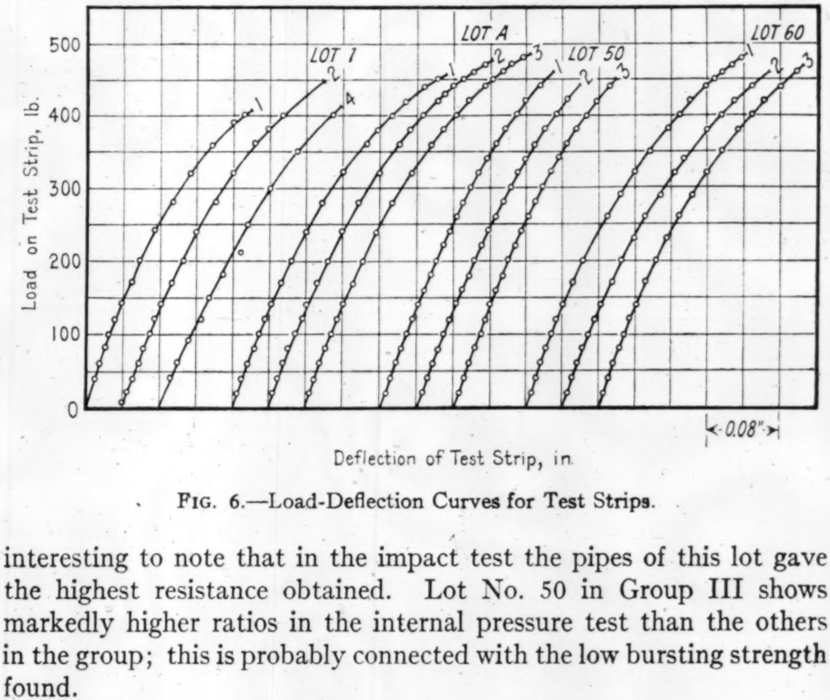

FIG. 6.—Load-Deflection Curves for Test Strips.

Information on the variations in strengths among the pipes of a given lot is given in the several tables under the heading of mean deviation. The numerical difference between the average strength of a lot and the value of each individual test piece was found and expressed in terms of the average strength. The average of these deviations for a given lot was calculated and thus represents the mean deviation from the average value of the lot. It has been expressed as a percentage of the average strength. The mean deviations recorded in Table II indicate that the pipes of the several lots as tested by internal pressure gave small variations in tensile resistance, even though there were generally only three specimens in a lot. It will be borne in mind that the tensile resistance reported is based upon the thickness of the pipe at the break. When there are three specimens in a lot the maximum deviation is, of course, one-half greater than the mean deviation. It will be noted that the average mean deviations in bursting strength for a group ranges from 4.4 per cent for Group II to 15 per cent for Group IV. The mean deviations for the flexure tests are given in Table III; the deviations are slightly less than for the bursting strengths. The mean deviations for the test specimens are given in Table VI.

TABLE VII.—SECANT MODULUS OF ELASTICITY.

Group	Lot	Secant Modulus of Elasticity, *E*, of Pipe, lb. per sq. in.	Secant Modulus of Elasticity, *E*, of Test Strip, lb. per sq. in.	Ratio *E* of Test Strip to *E* of Pipe	Secant Modulus of Elasticity, *E*, of Test Bar, lb. per sq. in.	Ratio *E* of Test Bar to *E* of Pipe	Secant Modulus of Elasticity, *E*, of Ring, lb. per sq. in.	Ratio *E* of Ring to *E* of Pipe
I	1	10 150 000	7 500 000	0.74	11 870 000	1.17	9 720 000	0.96
	4	9 400 000	7 200 000	0.77	11 300 000	1.20	8 450 000	0.90
	20	10 030 000	7 710 000	0.77	10 470 000	1.04	10 220 000	1.02
	30	9 130 000	6 630 000	0.73	11 100 000	1.21	9 230 000	1.01
	40	10 200 000	7 430 000	0.73	11 470 000	1.12	11 200 000	1.10
	70	10 070 000	7 160 000	0.71	....	....	....	....
	71	9 600 000	6 870 000	0.72	....	....	....	....
	72	9 030 000	6 610 000	0.73	....	....	....	....
	73	10 940 000	7 320 000	0.67	....	....	....	....
	Average	9 840 000	7 160 000	0.73	11 240 000	1.15	9 760 000	1.00
II	A	9 750 000	7 530 000	0.77	11 490 000	1.18	9 320 000	0.96
	B	10 520 000	7 950 000	0.75	10 610 000	1.01	9 610 000	0.91
	F	10 420 000	8 250 000	0.79	10 850 000	1.04	9 810 000	0.94
	75	9 920 000	7 670 000	0.77	....	....	....	....
	Average	10 150 000	7 850 000	0.77	10 980 000	1.08	9 580 000	0.94
III	50	14 780 000	11 220 000	0.76	....	....	13 000 000	0.88
	51	15 080 000	11 860 000	0.78	....	....	11 170 000	0.74
	52	14 860 000	11 170 000	0.75	....	....	13 100 000	0.88
	74	14 670 000	11 050 000	0.75	....	....	....	....
	Average	14 850 000	11 320 000	0.76	....	....	12 420 000	0.83
IV	60	10 630 000	7 340 000	0.69	....	....	10 930 000	1.03
	76	10 260 000	7 560 000	0.74	....	....	....	....
	Average	10 450 000	7 450 000	0.72	....	....	10 930 000	1.03

In Fig. 5 are given sample load-deflection diagrams from the flexure tests of the pipe; they are representative of the results for several groups. In Fig. 6 are load-deflection diagrams from the flexure tests of test strips.

In Table VII is recorded the secant modulus of elasticity calculated from the data of the flexure tests of the pipe and from the auxiliary test specimens at the maximum load. For the flexure test of the pipe the formula $E = \frac{Pl^3}{48\ Iy}$, was used where E is the modulus of elasticity, P the maximum load on the pipe, l the span length, y the center deflection at load P, and I the moment of inertia of the section of the pipe, which was obtained in the manner already described for the flexure test. For the test strip, with third-point loading, the formula was $E = \frac{23\ Pl^3}{1296\ Iy}$, for the test bar $E = \frac{Pl^3}{48\ Iy}$, and for the ring $E = 0.15 \frac{Pr^3}{Iy}$, r being the radius of the ring. It will be noted that the use of these formulas in this way, of course, is empirical, but the values of E so found will allow comparisons of the stiffness of the various groups to be made. The number of specimens used in determining E is the same as those given in Tables II, III and VI.

It is seen from Table VII that the values of the modulus of elasticity in any group of pipe are fairly uniform for each of the forms of test specimen. From the flexure test of the pipe, an average of 9,840,000 lb. per sq. in. was obtained in Group I, with a mean deviation per lot of 5.0 per cent from the average, and a maximum deviation of 11.2 per cent; in Group II an average of 10,150,000 lb. per sq. in., with a mean deviation of 3.2 per cent and a maximum of 3.9 per cent; and in Group III an average of 14,850,000 lb. per sq. in., with a mean deviation of 0.8 per cent and a maximum of 1.6 per cent. From the flexure test of the test strips the averages are in Group I, 7,160,000 lb. per sq. in., with a mean deviation per lot of 4.2 per cent and a maximum deviation of 7.7 per cent; in Group II, 7,850,000 lb. per sq. in., with a mean deviation of 3.2 per cent and a maximum deviation of 5.1 per cent; and in Group III, 11,320,000 lb. per sq. in., with a mean deviation of 2.3 per cent and a maximum deviation of 4.8 per cent. The values obtained from the test bars are more variable. As only one ring for each lot was tested, a comparison can not be made; besides, a varying relation between stress and strain for irons of different quality will affect the use of the formula for modulus of elasticity more in the ring test than in beam tests.

The averages of the values of the modulus of elasticity and the variations for different lots indicate, as was to be expected, that for any of the groups the material in the different lots has quite similar stiffness characteristics. The ratios of the modulus of elasticity obtained by the test of the test strip to that by the test of the pipe have relatively little variation in the several groups (see Table VII), and thus it may be accepted that the average ratio for any group can be used for the purpose of judging of the quality of the pipe when test strips are available. The ratio, 0.75, seems to be representative of Groups I, II and III. With such agreement in the results, it is evident that the data of the test strip may be taken to represent the stiffness qualities of the pipe.

The secant modulus here used disregards the curvature of the load-deflection curve. The resilience is, of course, dependent on the form of the load-deflection curve. A study of the curves indicates that an approximation to the character of the curvature may be had through the use of the secant modulus of elasticity at half the maximum load. For the flexure tests of pipe, the curves examined show that the ratio of the secant modulus of elasticity for a point at half the maximum load to the value of the secant modulus at the maximum load ranged from 1.18 to 1.33 for Group I, 1.22 to 1.40 for Group II, and 1.10 to 1.15 for Group III. For the test strips the corresponding ratios average 1.42 for Group I, 1.49 for Group II, and 1.30 for Group III. The relative excess of the full resilience (found from the area of the load-deflection curve) over that determined from the

TABLE VIII.—CHEMICAL ANALYSIS OF CAST-IRON PIPE AND TEST BARS.

Constituents are given in percentages by weight.

Group	Lot	Number of Pipe	Number of Test Bars	Silicon, per cent	Sulfur, per cent	Manganese, per cent	Phosphorus, per cent	Graphitic Carbon, per cent	Combined Carbon, per cent	Total Carbon, per cent
I	1	1	..	1.38	0.050	0.35	0.80	3.05	0.62	3.67
	1	..	6	1.37	0.055	0.34	0.80	2.96	0.70	3.66
	4	1	..	1.38	0.075	0.34	0.77	2.80	0.77	3.57
	4	..	2	1.42	0.065	0.34	....	2.78	0.76	3.54
	20	1	..	1.56	0.131	0.58	0.72	2.83	0.81	3.64
	20	..	3	1.54	0.085	0.42	....	2.87	0.72	3.59
	30	1	..	1.63	0.070	0.44	0.66	2.83	0.77	3.60
	30	..	3	1.61	0.066	0.46	....	2.89	0.67	3.56
	40	1	..	1.67	0.088	0.57	0.49	2.75	0.76	3.51
	40	..	3	1.64	0.072	0.42	....	2.74	0.75	3.49
II	A	1	..	1.45	0.065	0.26	0.77	2.69	0.82	3.51
	A	..	6	1.41	0.079	0.30	....	2.64	0.91	3.55
	B	1	..	1.47	0.061	0.32	0.74	2.80	0.76	3.56
	B	..	8	1.40	0.069	0.34	....	2.78	0.80	3.58
	F	1	..	1.41	0.057	0.28	0.74	2.88	0.78	3.66
	F	..	5	1.40	0.067	0.34	....	2.71	0.82	3.53
III	50	2	..	1.97	0.053	0.34	0.81	3.43	0.09	3.52
	51	2	..	1.66	0.059	0.37	0.81	3.46	0.12	3.58
	52	2	..	1.88	0.052	0.38	0.80	3.48	0.08	3.56
IV	60	2	..	1.65	0.071	0.30	0.80	2.85	0.68	3.53
V	2	1	..	2.09	0.050	0.52	0.84	2.95	0.58	3.53
	2	..	3	2.06	0.052	0.48	....	2.85	0.67	3.52
	3	1	..	1.50	0.056	0.50	0.74	2.65	0.75	3.40
	3	..	3	1.47	0.061	0.48	....	2.61	0.77	3.38
	5	1	..	1.37	0.063	0.33	0.80	2.75	0.75	3.50
	5	..	2	1.36	0.058	0.30	....	2.78	0.76	3.54
VI	C	1	..	1.44	0.060	0.40	0.71	2.52	0.79	3.31
	C	..	9	1.55	0.083	0.39	....	2.52	0.77	3.29
	D	1	..	2.24	0.055	0.35	0.73	2.82	0.53	3.35
	D	..	8	2.24	0.058	0.36	....	2.76	0.59	3.35
	E	1	..	1.42	0.070	0.36	0.71	2.52	0.85	3.37
	E	..	7	1.43	0.094	0.40	....	2.58	0.76	3.34

triangular area under the secant modulus line is about two-thirds of the amount that the ratios named above exceed unity; thus, for the ratio 1.18 the full resilience will be 12 per cent greater than that derived by the use of the secant modulus line at maximum load, and for the ratio 1.33 the full resilience will be 22 per cent greater.

In Table VIII are given the results of chemical analyses of samples taken from pipe and test bars of each group, as furnished by Mr. J. T. MacKenzie, Chief Chemist of the American Cast Iron Pipe Co. The table is given in the thought that it may be of interest to some readers.

The flexure test, impact test and internal pressure test have merit in giving information on the strength properties of cast-iron pipe. It is believed that an impact test can be devised that will be helpful in getting at the minimum thickness allowable in pipe made by a given method of manufacture and useful in determining quality of metal and general degree of uniformity. The internal pressure test is a most searching test (the ordinary proof-pressure test gives relatively little information) and measurements of thickness of the broken pipe and auxiliary test specimens taken from it for use in determining strength and stiffness qualities will also give important information in judging of the quality of the output. The auxiliary test specimens may also be taken from a pipe that has been tested in other ways or even may be cut from pipe rejected because of a defect or imperfection that leaves most of its length free from such imperfections. Of these auxiliary test specimens the test strip has advantages over the others. It can be cheaply and quickly cut to exact dimensions throughout the length and without danger of breakage by means of a double cutter in a milling machine, the method used in making the test strips in this investigation. The beam depth of this test strip is uniformly $\frac{1}{2}$ in. and the width covers the full thickness of the wall of the pipe and thus the fibers across the width of the beam at any depth are equally stressed and the results may be taken as representative of the material of the wall. The two-point loading gives high stress over a greater length than would a load at midspan and thus is more searching. The relations of the strength properties of this specimen to the internal pressure strength and the flexural strength of the pipe are fairly uniform for most of the lots of pipe tested, and a ratio between modulus of rupture of test strip and internal pressure strength or flexural strength of full-size pipe may be established which with suitable tolerances may be put into specifications. The requirements for stiffness quality may also be specified through the modulus of elasticity of the test strip. In all these ways the test strip has qualities that make it a very useful auxiliary test specimen.

No attempt has been made to outline requirements for pipe of any group; nor has any effort been made to bring out what tests and inspection should be undertaken to detect variations and lack of uniformity in thicknesses and the presence of flaws, defects, and unsatisfactory material in pipes that have not been subjected to the tests outlined, or to avoid improper practices in manufacture. Instead, the relations between the various tests and properties have been considered.

The following manufacturers of cast-iron pipe contributed the funds for the investigation from which most of the data given in this paper have been taken: Glamorgan Pipe and Foundry Company, Lynchburg Foundry Company, Donaldson Iron Company, Warren Foundry and Pipe Company, R. D. Wood and Company, and American Cast Iron Pipe Company.

DISCUSSION

Mr. MacKenzie.

MR. J. T. MACKENZIE.[1]—The test strip as here proposed by Messrs. Talbot and Richart is by far the best test yet devised for the metal in a cast-iron pipe. The use of strips heretofore has been handicapped because (1) the depth of the beam depended on the thickness of the pipe, which was a very large variable; (2) the test was on only one surface of the pipe wall; (3) the curvature had a pronounced effect on the moment of inertia as between tests in the convex or concave position; (4) the rough surface was difficult to measure within the accuracy required for this dimension; and (5) the load concentrated at one point was not sufficiently searching. Mr. Talbot, by his ingenious idea of keeping the depth constant and splitting the load, has eliminated the major objections enumerated above and reduced the others to a point where they cease to be of importance.

There is one viewpoint which the authors have merely touched upon in their paper that is worthy of more extended discussion and that is the resilience, and the present discussion is chiefly to develop this idea.

In the accompanying Table I, the results of the investigation are arranged in groups as used by the authors. Column 1 shows the tensile strength of the pipe wall, calculated from the bursting pressure. These figures seem low to anyone accustomed to vicarious test pieces, but they are easily understood when it is realized that the water pressure is applied to every point of the pipe wall. These results are not the results of the accidents of selection of test pieces, but are invariably the measure of the weakest place of the structure, and as such are truly indicative of the ability of the several products to withstand static pressure in the line, although they do not indicate ability to function under other stresses, such as bending or shock, which stresses are of prime importance. Mean deviations are shown in column 2.

Column 3 shows the results of the impact test, calculated as height of drop in feet divided by the square of the average wall thickness in inches. This method is chosen because the tests indicate that the resistance to impact varies quite closely as the square of the wall thickness. This test was designed to show the relative ability of the

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[1] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

TABLE I.—SUMMARY OF RESULTS OF INVESTIGATIONS ON CAST-IRON PIPE.

Lot	Internal Pressure Tests		Impact Test	Flexure Tests of Pipe			Flexure Tests of Strips	
	Bursting Strength, lb. per sq. in.	Mean Deviation, per cent of average strength for each lot	Height of Drop, ft., divided by Square of Average Thickness, in.	Modulus of Rupture, lb. per sq. in.	Mean Deviation, per cent of average strength for each lot	Secant Modulus of Elasticity, lb. per sq. in.	Modulus of Rupture, lb. per sq. in.	Secant Modulus of Elasticity, lb. per sq. in.
	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*
GROUP I.—VERTICALLY CAST, SAND MOLD								
1	13 900	4.8	16.7	29 000	3.8	10 150 000	35 300	7 500 000
4	16 100	10.3	16.6	32 200	6.4	9 400 000	38 300	7 200 000
20	14 700	9.2	14.0	27 000	2.2	10 030 000	33 900	7 710 000
30	13 700	2.6	15.3	26 800	0.7	9 130 000	33 700	6 630 000
40	13 400	15.6	20.3	29 300	7.1	10 200 000	39 100	7 430 000
70	14 800	3.9	14.4	28 400	5.3	10 070 000	35 000	7 160 000
71	14 600	9.3	16.0	26 300	2.7	9 600 000	29 300	6 870 000
72	14 400	3.7	18.2	26 900	6.5	9 030 000	32 300	6 610 000
73	16 400	7.3	14.2	26 800	4.9	10 940 000	35 500	7 320 000
Average	14 700	7.4	16.2	28 100	4.4	9 840 000	34 700	7 160 000
Full Resilience "Factor"[1]				95			207	
GROUP II.—CENTRIFUGAL PROCESS, SAND MOLD								
A	20 200	1.9	16.4	39 500	2.4	9 750 000	43 500	7 530 000
B	20 800	6.5	19.0	30 700	3.5	10 520 000	41 100	7 950 000
F	19 600	3.5	16.1	37 500	4.4	10 420 000	43 000	8 250 000
75	20 600	5.7	18.1	38 600	8.5	9 920 000	46 000	7 670 000
Average	20 300	4.4	17.4	36 600	4.7	10 150 000	43 400	7 850 000
Full Resilience "Factor"[1]				158			316	
GROUP III.—CENTRIFUGAL PROCESS, METAL MOLD								
50	15 700	7.5	14.6	34 500	5.8	14 780 000	50 800	11 220 000
51	24 400	12.3	12.6	32 200	6.5	15 080 000	43 500	11 860 000
52	25 800	5.6	14.2	37 000	12.2	14 860 000	48 800	11 170 000
74	30 000	3.8	11.6	42 700	4.3	14 670 000	50 700	11 050 000
Average	24 000	7.3	13.2	36 600	7.2	14 850 000	48 500	11 320 000
Full Resilience "Factor"[1]				98			250	
GROUP IV.—HORIZONTALLY CAST, SAND MOLD								
60	15 600	19.3	13.1	33 100	8.1	10 630 000	40 000	7 340 000
76	20 900	10.6	16.4	36 100	4.7	10 260 000	43 700	7 560 000
Average	18 200	15.0	14.8	34 600	6.4	10 450 000	41 900	7 450 000
Full Resilience "Factor"[1]				137			306	

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[1] **These values are determined by dividing the square of the modulus of rupture by the modulus of elasticity, and correcting for curvature of the load-deflection curves as explained in the text. The units are purely arbitrary (pounds divided by inches^2), but the values are relative and, for each test, comparable.**

Mr. MacKenzie.

several groups to stand the blows of loading, shipping and laying; and it is, at least, some measure of the ability to withstand water hammer.

Columns 4, 5, and 6 show the results of the flexure tests on the full length pipe tested as a beam. This test shows the value of the pipe in case of ground movement, or any other bending stresses. The values for modulus of elasticity show inversely the bending qualities, that is, the lower the value of modulus of elasticity, the more the pipe will bend. Ability to bend is a far better quality in a pipe than ability to resist bending, as in many cases the force is practically irresistible for short distances, and the only hope for the pipe is for it to yield. Tensile strength, as indicated by the modulus of rupture, is desirable if flexibility is not sacrificed in its attainment, but of the two, flexibility is of much greater importance.

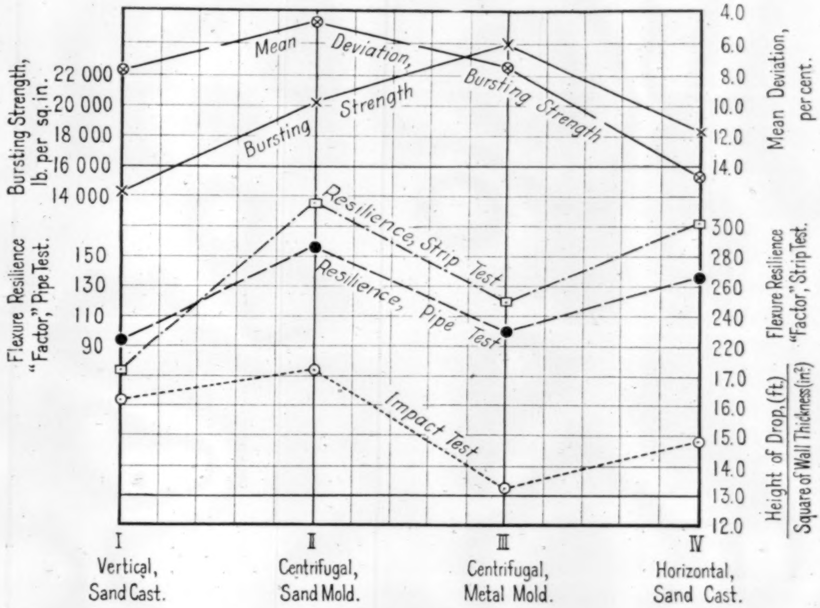

FIG. 1.—Resilience, Bursting Strength and Impact Test Values of Cast-Iron Pipe.

Columns 6 and 7 show the moduli of rupture and of elasticity, calculated from the strip test as described by the authors, but placed by them in separate tables. Our purpose in assembling these data anew is to consider them collectively in their relation to the quality of the pipe as shown by the three principal tests.

Mr. MacKenzie. Just as elongation and reduction of area are given equal importance with tensile strength in considering the value of steel for structural use, the deflection of cast iron is considered of equal importance with the strength in tests of this material. With the different wall thicknesses involved the only way to express unit deflection is by means of Young's modulus of elasticity which, as the authors say, is not an absolute but a relative quantity. The resilience, or capacity of a beam for storing energy, is, for a standard beam, the area below the load-deflection curve, and if this were a straight line the resilience would be the product of the load and the deflection. A linearly relative quantity in this case is the square of the modulus of rupture divided by the modulus of elasticity, and since the span lengths are the same for all groups this quotient gives an accurate comparison of the work done in breaking the several specimens, when modified by the percentages given in the authors' discussion of resilience which correct for the curvature of the load-deflection curves. In the table this value is placed below the average for each group, and in Fig. 1 these results are shown graphically with the results for the drop test, the tensile strength from the bursting pressure, and the mean deviation in bursting strength for each group.

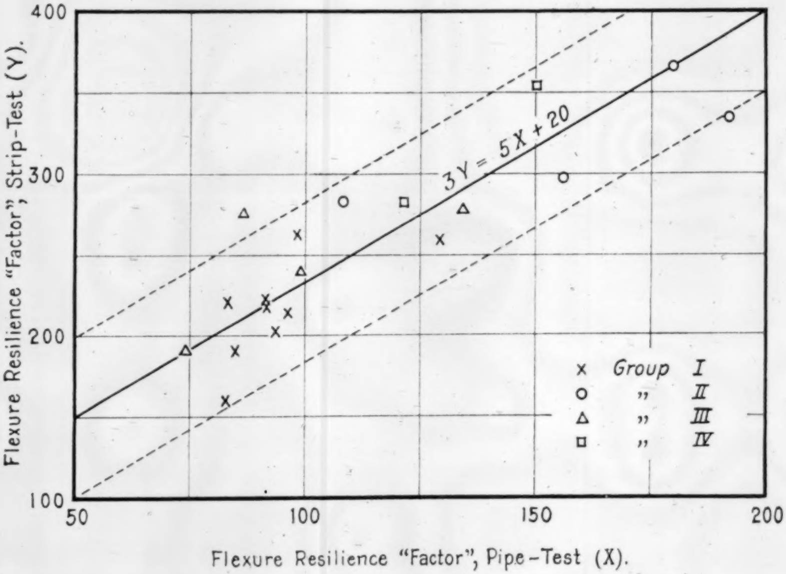

FIG. 2.—Relation of Flexure Resiliences as Obtained in Strip and Pipe Tests.

Mr. MacKenzie.

The agreement of the resilience curves for the flexure tests of strips and pipe is remarkable, and the drop test shows the same general relations for the several groups.

In Fig. 2 the resilience "factors" for each lot computed as above indicated, are plotted for the flexure tests of pipe and strips. The units used in both cases are arbitrary and are not at all the same, but the values are relative and, for each test, comparable. As both tests are based on rather small areas of the pipe wall and only a few specimens, it is quite remarkable that only one lot shows more than 25 per cent variation from an average line as shown, having the equation: $3Y = 5X + 20$.

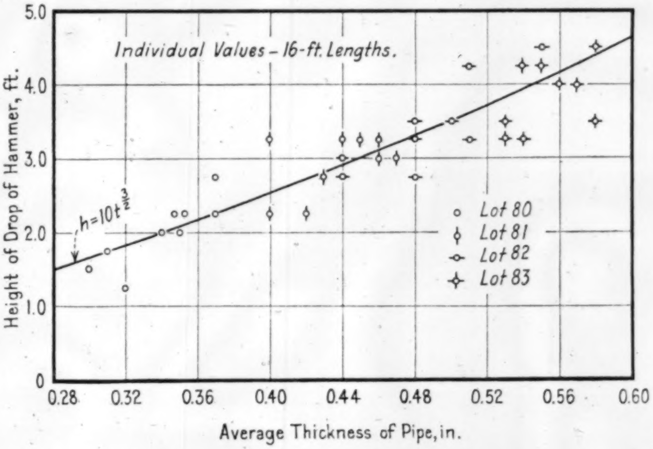

FIG. 3.—Relation of Thickness of Wall and Height of Drop in Impact Test with Supports 2 ft. Apart on Pipe of 16-ft. Length (Individual Values).

Mr. Green.

MR. A. W. F. GREEN.[1]—I have just a few questions to ask. The first is, what is the limit of tensile strength for good quality pipe? Another one, why is the impact test better than destruction by internal pressure as a test for good pipe? Another is, is the bursting strength the right criterion of a pipe's ability to resist water hammer?

Mr. Talbot.

MR. A. N. TALBOT (*Author's Closure*).—As noted in the paper, the impact testing machine was later modified by making the distance between centers of supports 2 ft. instead of 10 ft. and arranging it so as to permit testing a pipe at several points along its length. Tests were then made on pipe near the bell end, spigot end, and middle of the length. In the accompanying Fig. 3 are plotted thickness of pipe at the section tested and the height of drop at failure in tests with the modified impact testing machine on four lots of pipe made

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[1] Sales Metallurgist, The John Illingworth Steel Co., Elkins Park, Philadelphia, Pa.

Mr. Talbot. in 16-ft. lenghts by the centrifugal process (sand mold) used for the lots in Groups II and VI. In general, the pipe in these lots varied from thin pipe in lot 80 to greater thickness in lots 81 and 82, and a still greater thickness in lot 83. Three pipes of each lot were tested, each pipe at middle, bell end, and spigot end, making 36 tests in all. The individual results of the tests are plotted in Fig. 3. There is little scattering of points. There is no marked difference apparent in the relation between thickness of pipe at the section tested and height of drop, whether the test is made at middle, bell end, or spigot end. The curve shown on the figure, $h = 10\,t^{\frac{3}{2}}$, represents the results fairly well, h being the height of drop in feet and t the average thickness of the pipe wall in inches at the section of test.

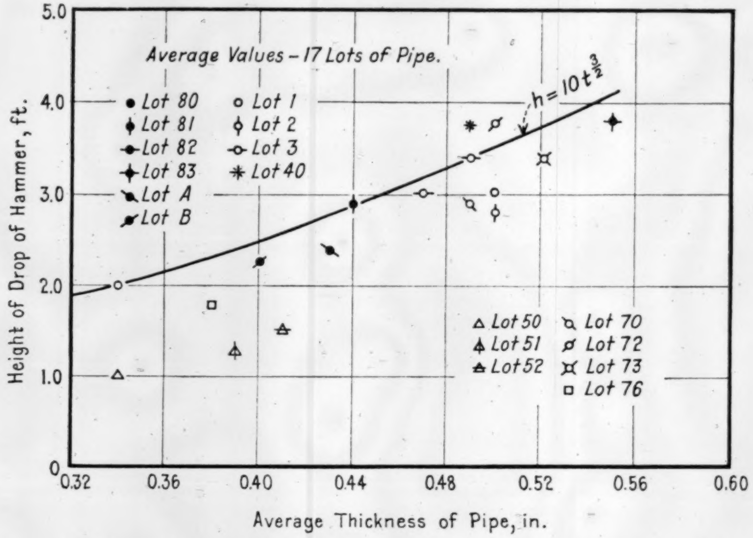

FIG. 4.—Relation of Thickness of Wall and Height of Drop in Impact Test with Supports 2 ft. Apart (Average Values for Each of Seventeen Lots).

In Fig. 4 are plotted the results of tests made on the remaining available pipes of the 13 lots named in the legend, together with those of the four lots already shown in Fig. 3. In lots 70, 72, 73, and 76 only a single pipe was tested; in lots 40, 50, and A, two pipes; and in the remaining, lots three pipes. In all cases, the points plotted are the averages of the results of the tests at middle and two ends for each given lot of pipe. The curve shown is the same as that in Fig. 3.

From a study of the action of the testing machine in the tests, it was concluded that the test in the new form avoids or reduces

Mr. Talbot. uncertainties involved in the inertia and deflection of the pipe and is more distinctly an impact test, and thus that it is an improvement on the original form of test.

Taking up the questions asked by Mr. Green: I wish he would answer them. They are good questions. It would be well to discuss them, though probably we have not enough information to answer. His first question was, what is the limit of the tensile strength of cast iron for a good quality of pipe? What is the limit of strength of wrought iron for a given purpose? What is the limit of strength of steel for a given purpose? Does the tensile strength tell all we need to know about wrought iron or steel? If so, why do we determine stretch? Why have we so often used the cold bend test? Why do we try in various ways to get information bearing on the properties of a material? Is a single property of cast iron all sufficient? And the second question is, why not the internal pressure test alone to the exclusion of the impact test? I do not myself know yet just what the value of an impact test is, but it is true that we need to know something about the toughness of a material; we must keep within proper limits of toughness, and possibly toughness may have bearing upon the permissible tensile strength of the cast iron. We generally also want to know something about the workability of the resulting metal. As to the relation between the internal pressure test and the resistance to water hammer, that again is uncertain. Perhaps Mr. Green is able to give information on that. Surely those properties which go to give toughness and resilience in internal pressure tests will also give resistance against water hammer.

FATIGUE OF METALS BY DIRECT STRESS

By P. L. Irwin[1]

Synopsis

This paper gives a comparison of the endurance limits obtained by flexural stress with those of direct stress for materials having lower proportional limits than endurance limits. Such a comparison under the reverse conditions is given in a paper presented by the author in 1925.[2]

Introduction

The present paper is supplementary to that presented in June of last year and deals with the same general subject: the comparison of endurance limits under flexural stress with those under direct stress (tension-compression).

The five materials discussed in the earlier paper were selected from those having proportional limits in tension above their endurance limits. In such cases the flexural-stress endurance limit was calculated by means of the usual beam formula, since the relation between stress and strain for these cases was linear. We were not concerned with this relation in computing the endurance limit obtained by direct-stress fatigue because the stress in the test specimen is uniform and equal to the load divided by the cross-sectional area. Hence, for the materials used in our first investigation, the agreement of the direct and flexural endurance limits was to be expected.

For materials whose proportional limit is below their endurance limit, however, it would hardly seem reasonable to expect the usual beam formula to apply in the computation of the flexural endurance limits, and a difference between the direct and flexural endurance limits would not be surprising. In order to determine whether such a difference exists, the materials given in the present paper were selected from those having proportional limits lower than their endurance limits.

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[1] Mechanical Engineer, Research Department, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] P. L. Irwin, "Fatigue of Metals by Direct Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 53 (1925).

PROCEDURE

The materials selected for this work were annealed electrolytic copper, annealed monel metal and annealed nickel. The chemical composition and heat treatment of the material is given below:

CHEMICAL ANALYSIS

	COPPER (ANNEALED) 12	NICKEL (ANNEALED) M-111	MONEL METAL (ANNEALED) M-110
Iron, per cent	0.010	0.80	2.16
Carbon, per cent	....	0.124	....
Nickel, per cent	....	97.03	68.21
Silicon, per cent	....	0.127	....
Manganese, per cent	....	1.24	1.99
Phosphorus, per cent	....	0.015	....
Sulfur, per cent	....	0.040	....
Copper, per cent	99.92	0.355	27.40
Zinc, per cent	Trace	....	....
Lead, per cent	0.012	....	....
Tin, per cent	0.010	....	....
Cobalt, per cent	....	0.27	0.19

HEAT TREATMENT

ANNEALED ELECTROLYTIC COPPER: 1 in. round.
Heated to 600° C. in 1½ hours, held 1 hour.
Furnace cooled to 200–250° C. and quenched in water.
COLD DRAWN NICKEL: 1 by 1 in.
Annealed at 800° C. for 1½ hours.
Box cooled.
HOT-ROLLED MONEL METAL: 1 in. round.
Heated to 750° C. in salt bath.
Held 1½ minutes and quenched in salt lime cyanide solution.

From each material, tension, flexural fatigue, and axial fatigue test specimens were machined. In the case of copper, test specimens were made for both beam and cantilever flexural fatigue.

TEST RESULTS

The results of the tension tests and of the fatigue tests are given in Tables I and II, respectively.

The proportional limit in tension of annealed copper was found to be 1875 lb. per sq. in. Referring to Fig. 1, note that the fatigue curve of the beam type test specimen gives an endurance limit of 11,300 lb. per sq. in. and that the cantilever type gives 10,100 lb. per sq. in. Taking into consideration the open scale used in plotting the ordinates, it will be apparent that the curves of Fig. 1 are quite smooth and that the difference in the endurance limits obtained is not as large as a cursory inspection would indicate. It must be remembered that both of these curves are obtained from flexural-stress test specimens.

In order to bring out more clearly the fact that a material may be subjected to millions of cycles of stress at a stress value of five times its proportional limit, the curves of copper shown in Fig. 2 are included. Both curves are plotted to the same stress scale and bring out this point more clearly than tabulated values of proportional limit and endurance limit.

The copper test specimens for direct stress were made unusually large in order to utilize loads of a magnitude which could be accurately applied in the Haigh type of testing machine. The diameter of the test specimen in the reduced mid-section was machined to 0.298 in. whereas the nickel and monel metal, being much stronger, were machined to 0.196 in. diameter. This heavy-body section of the copper test specimen resulted in a marked decrease in the proportional difference between the area at the button head and the area at the mid-section. The abrupt change in section at the button head caused a high stress concentration at this plane and brought about fracture of the specimen at the head. The direct stress points in Fig. 1 are indicated as unbroken in view of the fact that the test section had not fractured at the number of cycles indicated. The ferrous materials given in the author's paper of 1925 showed the tendency of

Table I.—Results of Tension Tests.

	Copper (annealed)	Nickel (annealed)	Monel Metal (annealed)
Proportional Limit, lb. per sq. in.	1 875	16 000	14 000
Yield Point, lb. per sq. in.	....	30 000	38 300
Tensile Strength, lb. per sq. in.	31 200	82 750	90 600
Elongation, per cent	60.0	50.9	46.8
Reduction of Area, per cent	69	65	69
Brinell Hardness Number	47	117	146

Table II.—Results of Fatigue Tests.

	Copper (annealed)	Nickel (annealed)	Monel Metal (annealed)
Endurance Limit, lb. per sq. in.:			
Flexural Cantilever	10 100	38 000	40 000
Flexural Beam	11 300	....	....
Direct Stress	11 000[a]	41 500	40 000[a]
Ratio, Endurance Limit to Tensile Strength	0.33	0.46	0.44

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[a] Approximately.

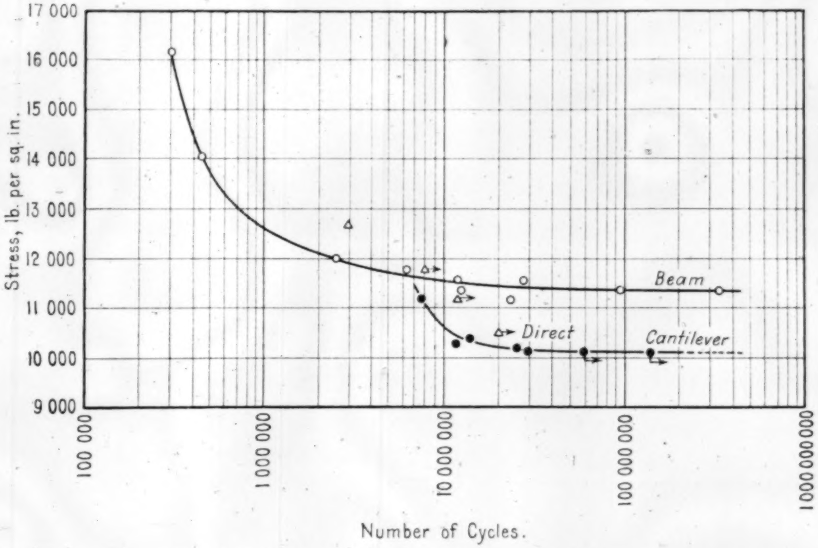

FIG. 1.—Endurance Curves for Annealed Copper.

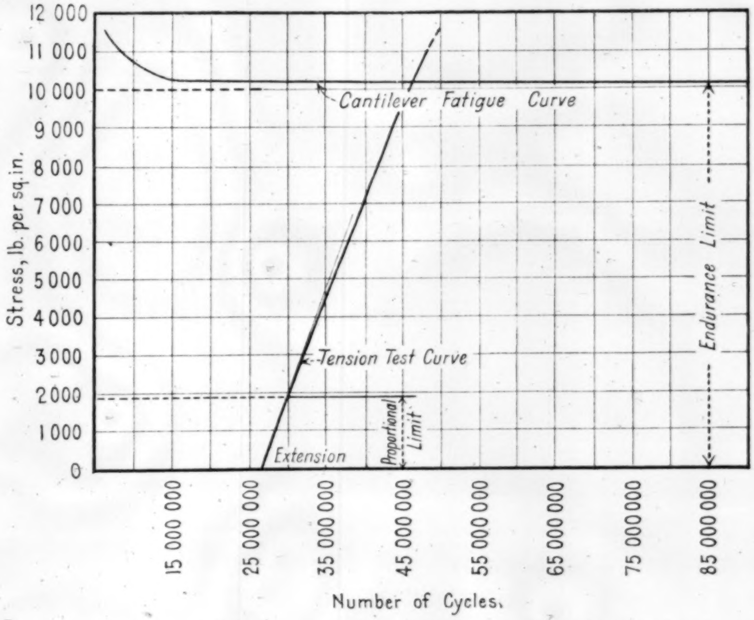

FIG. 2.—Comparison of Tension and Cantilever Fatigue Test Curves for Annealed Copper.

direct-stress fatigue curves to become asymptotic to the X-axis at fewer cycles than the flexural-stress endurance curves.[1]

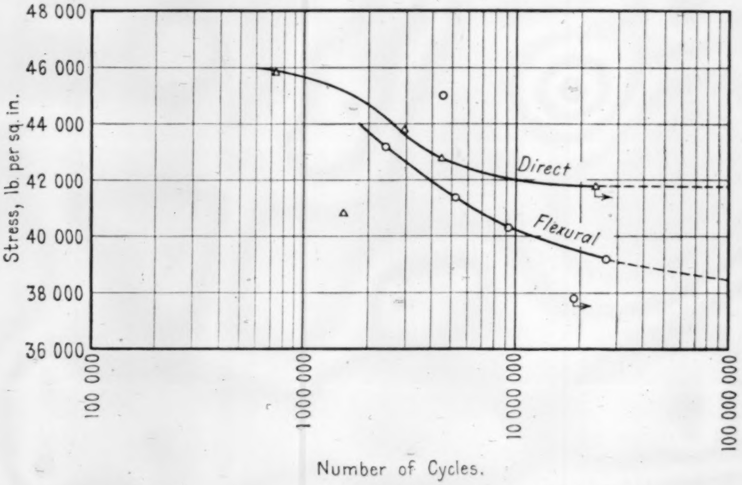

FIG. 3.—Endurance Curves for Pure Annealed Nickel.

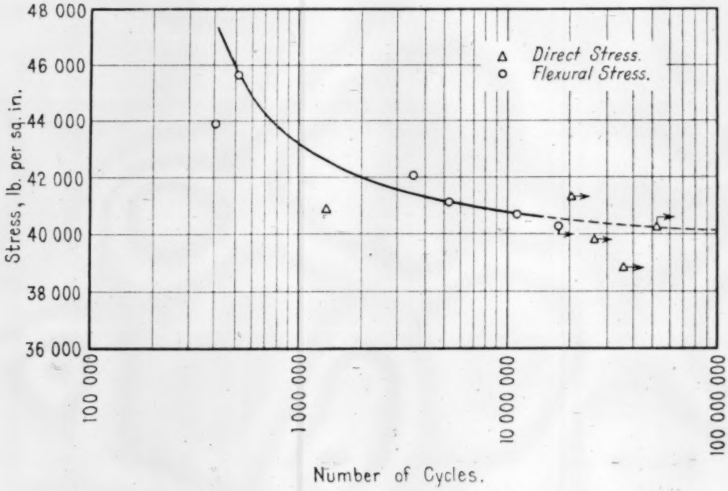

FIG. 4.—Endurance Curves for Annealed Monel Metal.

The nickel curves (Fig. 3) of the present paper show the same characteristic for a non-ferrous material. For this reason it seems reasonable to assume that the endurance limit by direct stress, for copper, is above that obtained by the cantilever flexural-stress curve. The object of this investigation being the comparison of direct-stress and flexural-stress endurance limits for materials of low proportional limit, it is felt that the direct-stress tests have indicated an agreement in endurance limits for the two types of stress. Further tests on properly filleted direct-stress test specimens are now in progress to check this conclusion.

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[1] D. J. McAdam, Jr., has pointed out the fact that this feature of the two curves is given by Gough in his book, "The Fatigue of Metals."

Due to the fact that we have but one direct-stress fatigue machine and since accurate endurance limits were not essential to the solution of our problem, some of the nickel and monel metal test specimens were removed earlier than would have been the case if time had permitted longer tests.

CONCLUSIONS

The general conclusion drawn from these tests is that the endurance limit of materials under direct stress is the same as that under flexural stress when the proportional limit in tension is *below* the endurance limit. Since the same conclusion was previously observed by the author for materials in which the proportional limit is *above* the endurance limit, we may say that the endurance limit of a material under direct stress is substantially the same as that of the same material under flexural stress regardless of the value of the proportional limit in tension.

Acknowledgment.—The author is indebted to Mr. S. M. Kintner, Manager of the Westinghouse Research Department, for permission to publish these results, to Mr. J. M. Lessells for his assistance in this work and to Mr. T. Hengstenberg, laboratory assistant.

[For Discussion on Fatigue of Metals, see page 269.—ED.]

STRESS-STRAIN-CYCLE RELATIONSHIP AND CORROSION-FATIGUE OF METALS[1]

By D. J. McAdam, Jr.[2]

Synopsis

Part I describes the material and the methods of investigation.

Part II discusses stress-strain-cycle relationship. The following subjects are considered: The stress-cycle graph extended to nominal stresses well above the tensile strength of the material and over a range from less than 1000 to more than 100,000,000 cycles; interrelationship between the stress-cycle, stress-deflection and deflection-cycle graphs; effect of cyclic repetition on deflection and hysteresis; the stress-cycle graph as affected by cycle frequency, thermal conductivity of specimen and environment, and form of specimen; extrapolation of stress-cycle graphs.

Part III discusses corrosion-fatigue. Evidence is presented that very slight corrosion when simultaneous with fatigue causes low resistance to fatigue. The damaging effect is greater the harder the steel. The advantage of heat treatment may thus be practically neutralized. Ratios of "corrosion-fatigue" limits to the endurance limit are compared with other physical properties of steel. The practical importance of corrosion-fatigue for ferrous and non-ferrous metals is discussed.

PART I.—OUTLINE OF INVESTIGATION, DESCRIPTION OF MATERIAL AND METHODS

Outline of Investigation

In recent publications[3,4,5] from the Naval Engineering Experiment Station there has been considerable discussion of the form of the stress-cycle graph. In the discussion of one of these papers,[4] it was evident that some readers considered the determination of the correct form of the stress-cycle graph of little importance. For the experimenter attempting to determine the endurance limit of a metal, however, it can be shown that a knowledge of the correct form of the graph is of great practical importance. Moreover, a knowledge of the form of the stress-cycle graph as affected by various factors must be the foundation of any satisfactory theory of fatigue of metals.

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[1] Published by permission of the Secretary of the Navy.

[2] Metallurgist, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[3] D. J. McAdam, Jr., "Endurance Properties of Metals," *Mechanical Engineering*, July, 1925.

[4] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part I," Paper 1506E, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, October, 1925.

[5] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, February, 1926.

In the above-mentioned papers it has been shown that on a semilogarithmic scale the lower portion of the stress-cycle graph, the portion ordinarily determined in endurance tests, is a curve with horizontal asymptote. Evidently, however, if the graph be extended upward beyond the portion usually included in endurance tests, the increase in slope could not continue indefinitely; the curve must either approach a sloping straight line or reverse its curvature.[1] It seemed desirable to investigate the form of the stress-cycle graph extended as far upward as possible and to investigate the influence of various factors on the form of the entire graph. An investigation of this subject at the Naval Experiment Station had not proceeded far, however, before it was found necessary to include stress-deflection and deflection-cycle graphs in the investigation and also to investigate corrosion-fatigue of metals.

TABLE I.—MECHANICAL TREATMENT OF MONEL METAL BARS.

Designation	Size of Bar, in.	Reduction of Area, Due to Cold-Working, per cent	Remarks
DY	$1\frac{1}{2}$ square	0	Hot-rolled annealed at 1400° F. (760° C.)
DYE	$1\frac{1}{2}$ "	0	As hot-rolled
DYA	$1\frac{3}{8}$ "	15	Cold-rolled
DYB	$1\frac{1}{4}$ "	30	Cold-rolled
DYC	$1\frac{1}{8}$ "	45	Cold-rolled
DYD	$\frac{15}{16}$ "	60	

MATERIAL

Monel metal for this work was furnished by the International Nickel Co. Hot-rolled bars and bars that had received various degrees of cold working were manufactured from the same heat. This material, as shown by metallographic examination, was of excellent quality and with few non-metallic inclusions. The unusually generous quantity of this material furnished by the manufacturers made it possible to extend the investigation far beyond the original plans.

Ingot iron was supplied for this investigation by the American Rolling Mill Co. The other materials used in this work were obtained by purchase from various companies. Details of mechanical treatment and heat treatment and the chemical composition are given in Tables I, II and III.

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[1] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, February, 1926.

The monel metal was supplied in the form of square bars of various sizes according to the amount of reduction in cold rolling. Table I gives details of mechanical treatment.

Each lot of steel was from the same manufacturer and from the same heat, with the exception of the stainless iron. As this latter material was from one source but from different heats, in Table II the range of values obtained in chemical analysis is given. Table II gives the chemical composition of the materials used. Table III gives details of the heat treatment made at the Naval Experiment Station.

TABLE II.—CHEMICAL COMPOSITION OF MATERIAL.

Each experiment station value represents two determinations.

Material	Designation	Carbon, per cent	Nickel, per cent	Copper, per cent	Iron, per cent	Manganese, per cent	Silicon, per cent	Phosphoru per cent	Sulfur, per cent	Lead, per cent	Chromium, per cent
Monel Metal, Cold Rolled	DYD	0.21	69.07[b]	28.04	1.60	1.02	0.03	0.017	0.017	[d]	.....
Monel Metal, Cold Rolled	DYC	0.21	69.07[b]	28.02	1.63	1.03	0.02	0.023	.....	[d]	.....
Monel Metal, Cold Rolled	DYB	0.21	69.02[b]	28.05	1.64	1.03	0.03	0.023	.....	[d]	.....
Monel Metal, Cold Rolled	DYA	0.21	69.10[b]	27.99	1.62	1.03	0.03	0.022	.....	[d]	.....
Monel Metal, Hot Rolled	DY	0.21	69.03[b]	28.03	1.63	1.03	0.03	0.024	0.016	[d]	.....
Average	...	0.21	69.06	28.02	1.62	1.03	0.03	0.022	0.017	....	.....
Ingot Iron	EF	0.024	....	.....	....	0.014	0.002	0.003	0.032	....	.....
Stainless Iron	ZZ (F-509)[c]	0.12	0.34	.....	....	0.28	0.13	0.017	0.023	....	11.58
Stainless Iron	ZZ (F-510)[c]	0.12	0.35	.....	....	0.32	0.15	0.016	0.018	....	11.85
Stainless Iron	ZZ (F-521)[c]	0.09	0.30	.....	....	0.37	0.19	0.019	0.018	....	12.35
Stainless Iron	ZZ (F-522)[c]	0.13	0.35	.....	....	0.37	0.16	0.017	0.016	....	12.20
Stainless Iron	ZZ[a]	0.09 to 0.13	0.30 to 0.35	.....	....	0.28 to 0.37	0.13 to 0.19	0.016 to 0.019	0.016 to 0.023	....	11.58 to 12.39
Carbon Steel	AG	0.362	....	.....	....	0.613	0.019	0.009	0.015	....	.....
Nickel Steel	CB	0.33	5.18	.....	....	0.49	0.40	0.017	0.013	....	.....
Chrome-Nickel Steel	BD	0.38	1.56	.....	....	0.66	0.32	0.024	0.017	....	0.80

[a] Material from different heats, extreme variations in composition are shown.
[b] By difference.
[c] Figures in parentheses refer to heat numbers. These values are manufacturer's results.
[d] None detected.

TABLE III.—HEAT TREATMENT.

Material	Designation	Heated to		Time Held, minutes	Cooled in	Reheated to		Time Held, minutes	Cooled in
		Deg. Fahr.	Deg. Cent.			Deg. Fahr.	Deg. Cent.		
Monel Metal, Cold Rolled	DYD-8	800	427	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYD-9	900	482	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYD-12	1200	649	60	Air	....	...	...	....
Monel Metal, Cold Rolled	DYD-14	1400	760	60	Air	....	...	...	....
Monel Metal, Cold Rolled	DYC-8	800	427	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYC-10	1000	538	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYB-8	800	427	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYA-8	800	427	180	Air	....	...	...	....
Monel Metal, Cold Rolled	DYA-10	1000	538	180	Air	....	...	...	....
Ingot Iron	EF-17.5	1750	954	45	Furnace	....	...	...	....
Carbon Steel	AG-W-9	1550	843	60	Water	900	482	120	Furnace
Carbon Steel	AG-15.5	1550	843	60	Furnace	....	...	...	....
Nickel Steel	CB-W-10	1450	788	60	Water	1000	538	120	Furnace
Chrome-Nickel Steel	BD-W-10	1500	816	60	Water	1000	538	120	Furnace
Chrome-Nickel Steel	BD-W-11	1500	816	60	Water	1100	593	120	Furnace
Chrome-Nickel Steel	BD-W-12	1500	816	60	Water	1200	649	120	Furnace
Chrome-Nickel Steel	BD-15	1500	816	60	Furnace	....	...	...	....

TABLE IV.—TENSION TESTS.

Material	Designation	Condition	Tensile Strength, lb. per sq. in.		Johnson's Limit, lb. per sq. in.		Proof Stress, lb. per sq. in.		Elastic Limit, lb. per sq. in.		Proportional Limit, lb. per sq. in.		Elongation in 2 in., per cent		Reduction of Area, per cent	
			Average	Mean Deviation	Average	Mean Deviation	Average	Mean Deviation	Average	Mean Deviation	Average	Mean Deviation	Average	Mean Deviation	Average	Mean Deviation
Monel Metal, Cold Rolled	DYD	As received	139 300^{f}	±4 300	98 400	±3 100	81 400	±7 100	63 900	±5 000	57 100	±3 800	12.7	±0.2	58.3	±1.8
Monel Metal, Cold Rolled	DYD-8	Annealed	135 500^{h}	±1 200	97 100	±2 100	90 900	±2 000	75 800	±4 900	62 400	±2 900	21.1	±0.6	55.8	±1.5
Monel Metal, Cold Rolled	DYD-9	Annealed	132 200^{d}	±1 400	94 200	±600	92 500	±600	83 400	±1 900	67 400	±2 500	22.2	±0.4	59.8	±1.0
Monel Metal, Cold Rolled	DYD-12	Annealed	91 300^{b}	±700	41 500	±1 000	41 800	±1 200	40 000	±1 700	27 600	±6 500	47.8	±1.4	71.6	±0.8
Monel Metal, Cold Rolled	DYD-14	Annealed	88 500^{a}	±450	35 000	±5 000	36 000	±3 000	32 500	±5 000	25 500	±7 000	46.5	±0.5	71.8	±0.8
Monel Metal, Cold Rolled	DYC	As received	133 600^{f}	±2 700	101 600	±900	84 600	±4 300	74 100	±10 500	54 500	±2 400	12.8	±0.7	55.2	±3.7
Monel Metal, Cold Rolled	DYC-8	Annealed	128 000^{h}	±1 000	90 700	±2 200	85 300	±1 900	77 700	±1 600	72 500	±2 800	21.7	±0.4	57.2	±2.0
Monel Metal, Cold Rolled	DYC-10	Annealed	119 800^{a}	±1 250	81 000	±0	82 000	±0	73 500	±5 000	43 000	±2 500	26.5	±0.9	63.0	±1.1
Monel Metal, Cold Rolled	DYB	As received	128 000^{c}	±1 300	99 000	±2 000	90 800	±2 750	77 800	±3 650	70 500	±2 500	13.9	±0.6	61.1	±0.6
Monel Metal, Cold-Rolled	DYB-8	Annealed	122 400^{g}	±2 300	83 400	±4 900	80 200	±2 100	67 000	±6 100	62 100	±3 300	22.5	±1.6	61.5	±2.6
Monel Metal, Cold Rolled	DYA	As received	113 300^{e}	±700	85 100	±900	70 900	±6 000	55 600	±9 700	41 500	±4 700	18.6	±1.6	64.7	±1.5
Monel Metal, Cold Rolled	DYA-8	Annealed	107 900^{e}	±1 200	71 600	±2 600	70 700	±1 200	59 700	±3 000	45 000	±4 000	26.4	±0.9	66.8	±0.7
Monel Metal, Cold Rolled	DYA-10	Annealed	100 400^{b}	±600	65 200	±4 900	65 300	±3 100	59 300	±2 900	53 300	±1 100	31.8	±0.6	67.5	±0.6
Monel Metal, Hot Rolled, Annealed	DY	As received	87 700^{e}	±400	34 600	±1 100	35 700	±1 000	32 900	±1 900	26 100	±1 900	48.5	±1.1	71.2	±0.6
Monel Metal, Hot Rolled	DYE	As received	87 000^{g}	±200	32 800	±900	34 200	±1 000	31 800	±2 300	16 500	±2 000	40.9	±5.1	64.0	±2.6
Monel Metal, Hot Rolled	DYE-14	Annealed	86 700^{a}	±200	32 500	±500	33 800	±300	30 500	±500	19 300	±1 800	45.8	±1.8	68.3	±0.3
Ingot Iron	EF-17.5	Annealed	41 500^{c}	±500	15 500	±800	15 600	±800	12 000	±2 500	9 300	±1 000	48.8	±2.9	76.2	±2.7
Stainless Iron	ZZ	As received	108 500^{c}	±2 300	74 500	±800	76 600	±2 100	65 800	±2 900	58 800	±1 900	22.3	±1.1	67.8	±0.8
0.36-per-cent Carbon Steel	AG-W-9	Quenched and drawn	103 600^{b}	±1 600	75 700	±2 200	75 300	±2 900	74 700	±3 100	65 000	±5 300	25.0	±1.3	57.8	±2.0
0.36-per-cent Carbon Steel	AG-15.5	Annealed	79 200^{d}	±1 100	45 200	±2 200	45 400	±2 100	41 800	±7 300	35 000	±2 800	31.3	±0.8	48.7	±0.5
5-per-cent Nickel Steel	CB-W-10	Quenched and drawn	129 600^{g}	±1 700	119 900	±900	119 900	±2 000	116 000	±5 800	87 000	±3 500	21.8	±0.6	56.4	±1.2
Chrome-Nickel Steel	BD-W-10	Quenched and drawn	148 300^{k}	±3 620	138 900	10 000	129 500	±5 200	120 400	±10 320	92 100	±4 500	18.2	±1.3	54.0	±1.5
Chrome-Nickel Steel	BD-W-11	Quenched and drawn	132 900^{d}	±2 800	117 600	±3 100	117 900	±3 100	110 000	±8 000	85 000	±4 000	21.9	±1.1	57.4	±1.8
Chrome-Nickel Steel	BD-W-12	Quenched and drawn	115 800^{b}	±11 000	100 700	±900	101 300	±1 100	86 700	±14 400	64 300	±3 400	25.6	±1.5	61.3	±1.5
Chrome-Nickel Steel	BD-W-15	Annealed	105 800^{c}	±4 400	67 800	±1 750	62 800	±1 800	57 800	±5 750	46 500	±2 250	26.2	±2.4	52.2	±3.6

[a] Average of two determinations.
[b] Average of three determinations.
[c] Average of four determinations.
[d] Average of five determinations.
[e] Average of six determinations.
[f] Average of seven determinations.
[g] Average of eight determinations.
[h] Average of nine determinations.
[k] Average of ten determinations.

The cold-rolled monel metal was found to have high internal stress. The heat-treatment necessary to relieve this stress was investigated, but the results are omitted because of lack of space. Annealing for 3 hours at 800° F. (427° C.) was found to remove about half, and annealing for 3 hours at 1000° F. (538° C.) nearly all the internal stress, with relatively slight loss in strength.

Machines and Specimens

Endurance tests were made by the rotating-cantilever method described in previous papers.[1] The specimens used had conical taper so designed that the stress is nearly uniform for about 1½ in. of the specimen length, and the maximum stress is about ¾ in. out from the fillet.[1,2] The method of alternate transverse and longitudinal polishing has been previously described.[1,3] For the experiments described below, however, the specimens were finally given a transverse and longitudinal polish with alumina that had remained suspended in water for three hours.

Tension Test Results

Results of tension tests are given in Table IV. On account of lack of space only averages and mean departures from averages are recorded.

PART II.—STRESS-STRAIN-CYCLE RELATIONSHIP

The Stress-Cycle Endurance Graph

General Description of Fig. 1.—Stress-cycle endurance graphs for monel metal, in eight different conditions of mechanical treatment and heat treatment, are shown in Fig. 1. Details of composition, mechanical treatment, heat treatment, and physical properties may be found in Part I by use of the material designations on each graph of Fig. 1. On each graph are indicated by the short horizontal lines the tensile strength (T.S.), the tensile proof stress (P.S.), and the tensile elastic limit (E.L.). The graphs are arranged from left to right in order of increasing strength of material. The first two graphs are on the same scale. For each succeeding graph the horizontal scale is shifted one or two logarithmic intervals to the right. The horizontal scale is indicated by the figures along each graph. In relation to the horizontal scale the vertical scale is one-half that used in previous papers.

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[1] D. J. McAdam, Jr., "Endurance Properties of Alloys of Nickel and of Copper, Part I," *Transactions*, Am. Soc. Steel Treating, January, 1925.

[2] D. J. McAdam, Jr., "Endurance Properties of Metals," *Mechanical Engineering*, July, 1925.

[3] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, February, 1926.

Monel Metal Tested at 1400 r.p.m. in Air.—Results obtained with monel metal tested at 1400 r.p.m. in air are indicated in Fig. 1 by small open circles and the solid lines drawn through them. It was found possible to extend six of these stress-cycle curves to nominal stresses much higher than the tensile proof stress, even to stresses at which the specimens became bright red hot before fracture.

In a recent paper[1] a graph for hot-rolled monel metal was drawn with reversal of curvature near the upper end. The possibility of such reversal was indicated by the results there shown. The results given in Fig. 1 confirm the conclusions expressed in the previous paper, and show that reversal of curvature in the upper portion of the stress-cycle graph is not unusual. Evidently the lower portion of each graph is a curve with horizontal asymptote.

Monel Metal Tested at 1400 r.p.m. in Water Stream.—It seemed probable that the reversal of curvature, in the upper part of the graph for monel metal tested in air, is due in part to the above-described rise in temperature of the specimen. To eliminate or diminish the effect of this temperature variable on the stress-cycle graph, additional experiments were made with specimens on which a water stream impinged. In these experiments one or more diagonal streams of water at a temperature of 60 to 70° F. (15.6 to 21.1° C.) swept the revolving specimen from the outer to the inner fillet, so that the surface under stress was entirely surrounded by water. The results of these tests are shown in Fig. 1.

TABLE V.—RATIO OF ENDURANCE LIMIT TO TENSILE STRENGTH OF MONEL METAL.

Designation	Tensile Strength, lb. per sq. in.	Endurance Limit, lb. per sq. in.	Ratio, Endurance Limit to Tensile Strength
DY	87 700	36 000	0.41
DYA-10	100 400	40 000	0.40
DYA-8	107 900	42 000	0.39
DYB-8	122 400	47 000	0.38
DYC-10	119 800	50 000	0.42
DYC-8	128 000	51 000	0.40
DYD-9	132 200	54 000	0.41
DYD-8	135 500	54 000	0.40

The stress-cycle graphs for water-cooled specimens are represented in Fig. 1 by open triangles and by the broken lines passing through them. By use of these water-cooled specimens it was possible to extend the stress-cycle graphs upward to nominal stress greater than the tensile strength and in some cases greater than the actual tensile stress at maximum load. At the upper limits of such graphs the specimens usually broke at less than 1000 cycles; one specimen broke at only 300 cycles. For most of the materials represented in Fig. 1 the stress-cycle graphs for water-cooled specimens descend to a junction with the graphs for uncooled specimens. This was to be expected as the temperature of the water was practically the same as the temperature of the air. For the annealed material DY, however, the graph for water-cooled specimens in descending does not unite with the curve for uncooled specimens, at least within the range of cycles investigated. Reasons for this will be discussed below.

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[1] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, February, 1926.

Effect of Cold Working on Form and Position of the Stress-Cycle Graph.—As shown in Fig. 1 the horizontal asymptote (the endurance limit) is higher the greater the degree of cold working. The estimated endurance limits, with corresponding values for tensile strength and endurance ratio, are given in Table V. It will be noted that the endurance limit increases approximately in proportion to the increase in tensile strength due to cold working. These results are in agreement with previously expressed conclusions regarding the effect of cold working on the endurance limit. It is of interest to compare the endurance limit for material DY (Fig. 1) with that for material DYD-14 as obtained from Fig. 2. Material DY had been hot-rolled and annealed by the manufacturers at 1400° F. (760° C.). Material DYD-14 had been severely cold-rolled and afterwards annealed at the Naval Experiment Station at 1400° F. (760° C.). A comparison of the graphs for these two materials in Figs. 1 and 2 shows that the endurance properties of the material have not been injured by the severe cold working.

To show the effect of cold working on the form and position of the stress-cycle graph, some graphs from Fig. 1 have been assembled in Fig. 3. In this figure, the position of the scale is not shifted for individual graphs as in Fig. 1. It will be noted that with increase in the degree of cold working the graph is moved outward as well as upward. To determine the approximate position of the asymptote, therefore, the number of cycles must be increased with increase in the degree of cold working, unless a method of extrapolation be used. For comparison, a graph for a sample of hot-rolled monel metal, taken from a previous paper, is included in Fig. 3. This graph (AAX) is between the graph for fully annealed material and the graph for material DYA-8 that had been cold-rolled to a reduction of 15 per cent in cross-section. On the other hand, material DYE (illustrated in Fig. 7) which had been hot-rolled and not annealed has an endurance limit practically the same as that of fully annealed material. "Hot-rolled" material, therefore, differs in endurance properties from fully annealed material only in proportion to the amount of "cold working" applied in the hot-rolling process. The rate of curvature for the uncooled, fully annealed material is much greater than for even slightly cold-worked material. Reasons for this will be discussed below.

THE STRESS-DEFLECTION GRAPH

The Stress-Deflection Graph for Monel Metal.—As shown in Fig. 1, it was found possible to extend the stress-cycle graphs for water-cooled monel metal to nominal stresses well above the tensile strength of the material. This fact made it desirable to investigate the form of the stress-deflection graph for the type of specimen used. Results of this investigation are illustrated in Fig. 4. The graphs are arranged in order of decreasing degree of cold working and strength of material. On each stress-deflection graph is plotted the corresponding tensile stress-strain graph adjusted to the same initial slope. On each graph the designations P.L., E.L., P.S., J.L. and T.S. are used to indicate the tensile proportional limit, elastic limit, proof stress, Johnson's limit and tensile strength, respectively. The designation J.L.D. is used to indicate Johnson's limit for the stress-deflection graph.

As shown in Fig. 4, the stress-deflection graph continues upward in a nearly straight line to a nominal stress far above the stress at which the corresponding tensile stress-strain graph curves rapidly away from the line of initial slope. This difference between the stress-deflection and the tensile stress-strain graph is best indicated by a comparison of the position of the point J.L.D. with that of the corresponding point J.L.

A comparison of the graphs for the same material at differing cycle frequencies shows that the form of the graph is practically unaffected by cycle frequency. The indicated deflections are intended to be as nearly as possible initial deflections. Slight differences between graphs for the same material at differing frequencies, however, may be due to the fact that the higher the frequency the more difficult it is to obtain a measurement indicating the initial deflection.

For fully annealed material, DYE-14, one graph represents the stress-deflection relationship under practically static loading. There are also recorded hysteresis measurements for this same material. To save space, only half of each hysteresis loop is recorded. Each hysteresis loop represents the fifth cycle obtained at the corresponding stress. It was found that after about five cycles the loop for this material was sufficiently stable to permit measurement of the accuracy desired. In the hysteresis curve at the right of Fig. 4 the areas of these loops, expressed in inch-pounds per square inch, are plotted as abscissas. Investigation of hysteresis in relation to endurance properties is being continued.

The Stress-Deflection Graph for Steel.—For steel as for monel metal it was found possible to extend the stress-cycle graphs in Figs. 8 and 9 to nominal stresses well above the tensile strength of the material. As shown in Fig. 10, stress-deflection graphs for steels continue their original slope to nominal stresses far above the stresses at which the corresponding tensile stress-strain graphs curve rapidly away from their original line of slope. For steel as for monel metal the stress-(initial) deflection graph is not affected by cycle frequency.

Explanation of Difference in Form Between Stress-Deflection and Tensile Stress-Strain Graph.—The difference in form between the stress-deflection graph and the tensile stress-strain graph is evidently due to to the fact that the usual formula for calculating bending stress is not applicable in calculating stress above the elastic limit. In a specimen subjected to bending stress above the elastic limit the stress distribution is not that assumed in the formula. The actual tensile stress is lower than that assumed by the formula. In order to account, therefore, for the stiffness of the specimen loaded above its elastic limit, it seems probable that the maximum compressive stress is higher than that obtained from the formula. As the load on the specimen increases, the neutral plane probably descends below the axis of the specimen. The range of stress per cycle, therefore, in such a heavily loaded specimen is shifted toward the compression end and may be not much less than the range calculated by the usual formula.

THE DEFLECTION-CYCLE GRAPH

As illustrated in Fig. 1, the stress-cycle graphs for water-cooled specimens of severely cold-worked material show reversal of curvature at high stresses. As indicted by the short lines marked J.L.D. in Fig. 1, the point of reversal of curvature is near the point at which the corresponding stress-deflection graph in Fig. 4 begins to curve away from its original line of slope. It seemed desirable, therefore, to study the relationship between initial deflection and the number of cycles necessary to cause failure of water-cooled specimens tested at 1450 r.p.m. The relationship is illustrated in Fig. 5. As shown in this figure, the deflection-cycle graph is an asymptotic curve with no reversal at high deflections.

EFFECT OF CYCLE FREQUENCY ON THE STRESS-CYCLE GRAPH

Effect of Cycle Frequency on Upper Part of Stress-Cycle Graph for Monel Metal.—The fact that cycle frequency between 2 and 1800 r.p.m. has practically no effect on the form of the stress-(initial) deflection graph does not preclude the possibility that cycle frequency may greatly affect the form of the stress-cycle graph. Although the initial deflection is not affected by cycle frequency, it will be shown later that cycle frequency may influence the changes in deflection that occur during an endurance test. To investigate the effect of frequency on the stress-cycle graph, endurance tests were made with monel metal at frequencies ranging from 20 to 1450 r.p.m. The results are illustrated in Fig. 1.

Results obtained with material DYD-8 at 60 and 200 r.p.m. are represented in Fig. 1 by experimental points and a line intermediate between the lines representing cooled and uncooled material tested at 1450 r.p.m. The line for material tested at 200 r.p.m. has been extended downward far enough to show that it unites with the lines for material tested at 1450 r.p.m. The results for material tested at 60 and 200 r.p.m. evidently differ so slightly that no attempt was made to draw a separate line for each.

Results obtained with material DYC-8 at 20 and 50 r.p.m. are represented in Fig. 1 by lines descending to unite with the line for water-cooled material tested at 1450 r.p.m. Evidently with decrease in frequency from 1450 to 20 r.p.m. the stress-cycle graph for uncooled material approaches the position of the graph for water-cooled material tested at 1450 r.p.m. Evidently the graphs for 20 and 50 r.p.m. unite with the graph for water-cooled materials somewhat above its junction with the graph for uncooled material tested at 1450 r.p.m.

Results obtained with material DYB-8 at 20, 50, 200 and 700 r.p.m. are represented in Fig. 1 by experimental points and lines intermediate between the graphs for cooled and uncooled material tested at 1450 r.p.m. None of the specimens tested at frequencies other than 1450 r.p.m. were water cooled. With decrease in cycle frequency from 1450 to 20 r.p.m. the graph for uncooled material approaches the graph for water-cooled material tested at 1450 r.p.m. The various graphs for material DYB-8 in descending evidently unite to form a single line.

Evidently, therefore, for cold-rolled monel metal tested without artificial cooling, the slope of the upper portion of the stress-cycle graph increases with decrease in cycle frequency from 1450 to 20 r.p.m. Whether this increase in slope would continue with further decrease in cycle frequency is uncertain.

It seems probable that the above-described change in position of the stress-cycle graph with change in frequency is due chiefly to the accompanying change in rate of heat evolution. The temperature of the uncooled specimen becomes higher the greater the frequency. For this reason the graphs for material tested at low frequency could be extended much higher than the graphs for uncooled specimens tested at high frequency. The specimens tested at 20 r.p.m. became only slightly warm. Possibly, therefore, the difference in position between the graphs for uncooled material at 20 r.p.m. and water cooled-material at 1450 r.p.m. is due chiefly to difference in frequency alone. It may be assumed, therefore, that the effect of a range of frequency between 20 and 1450 r.p.m. for cold-rolled monel metal is chiefly the effect of the accompanying temperature changes. Since for monel metal the tensile strength decreases with increase in temperature, it might have been expected that the upper portion of the stress-cycle graph would be less steep the higher the cycle frequency.

Effect of Cycle Frequency on Upper Part of Stress-Cycle Graph for Steel.—Only a start has been made in investigating the effect of cycle frequency on the upper part of the stress-cycle graph for steel. Some results obtained with annealed 0.36-per-cent carbon steel, material AG-15.5, are recorded in Fig. 9. It will be noted that the upper portion of the graph for uncooled material tested at 1450 r.p.m. is above the graph for water-cooled material. The "temper colors" indicated on Fig. 9 show that the upper portion of this graph for uncooled material represents tests at temperatures as high as 600° F. The fact that mild steel at this temperature is stronger than at room temperature accounts for the steep slope of the graph. It will be noted also that the effect of cycle frequency on uncooled annealed mild steel is the reverse of its effect on monel metal. The slope of the graph increases with increase in frequency from 2 to 1450 r.p.m.

As shown in the same figure, the graph for water-cooled material tested at 1450 r.p.m. occupies an intermediate position. It is above the graphs for material tested at 2, 4, 20, 50 and 200 r.p.m. As the uncooled material tested at 2, 4 and 20 r.p.m. was only slightly warm, it seems probable that the difference in position between the graphs for these materials and the graph for water-cooled material is due not to temperature but chiefly to the effect of cycle frequency. The effect of cycle frequency at constant temperatures is apparently somewhat greater for annealed mild steel than for cold-rolled monel metal.

A few results obtained with quenched-and-tempered chrome-nickel steel, material BD-W-10, are recorded in Fig. 8. The upper portion of the graph for water-cooled material is above the corresponding portion of the graph for uncooled material tested at 1450 r.p.m. As indicated by the recorded "temper colors" the uncooled material reached temperatures as high as 600° F. (316° C.); finally it became red hot. At these temperatures evidently the material is weaker than at room temperature. The results obtained at a stress of 125,000 lb. per sq. in. and at 50 r.p.m. would indicate that the effect of cycle frequency alone between 50 and 1450 r.p.m. is slight for the upper portion of the stress-cycle graph for all except soft steels. The effect of cycle frequency on the lower part of the stress-cycle graph for metals will be discussed later.

EFFECT OF CYCLIC REPETITION ON DEFLECTION AND HYSTERESIS

Effect of Cyclic Repetition on Deflection and Hysteresis of Monel Metal.—The changes in deflection and in hysteresis due to cyclic repetition are greatly influenced by cycle frequency and by the rate of interchange of heat between specimen and surroundings. This is illustrated in Fig. 6 by representative deflection-cycle graphs selected from a large number of results obtained with monel metal. In this figure the lines represent the change of the deflection with cyclic repetition, and the designations at the end of each line represent the breaking point. Fig. 6 (*a*) represents results obtained at various stresses with uncooled specimens of material DYC-8 tested at 50 r.p.m. With increase in nominal stress, both initial deflection and slope of the graph increase.

In Fig. 6 (*b*) a comparison of the upper and lower curves shows the effect of stress variation. A comparison of the middle and lower curves shows the effect of cycle frequency. The difference between these two curves is due almost entirely to difference in the rate of increase in temperature at the two different cycle frequencies.

In Fig. 6 (*c*) a comparison of the two intermediate curves shows the effect of cycle frequency. This effect is due to difference in rate of increase in temperature at the two different frequencies. The lower three curves would start from the same point, if it were possible to obtain specimens identical in strength and dimensions. The effect of rate of heat interchange between specimen and surroundings is illustrated by a comparison of the two intermediate curves with the lower curve. Although the lower curve represents a test at much higher frequency than the other two, this curve is flatter because the greater rate of heat evolution is more than compensated by the greater rate of heat removal from this water-cooled specimen.

Fig. 6 (*d*) presents additional illustration of the influence of rate of heat evolution and removal on the slope of the deflection-cycle graph.

As the variation in hysteresis is qualitatively the same as the variation in deflection, it is evident that with cyclic repetition of these highly stressed specimens hysteresis increases. Increase in hysteresis means increase in rate of heat evolution. If the rate of heat evolution is greater than the rate of heat removal, the temperature rises and the rise in temperature accelerates the rate of increase hysteresis.

Effect of Cyclic Repetition on Deflection of Steel.—Figs. 11 (*a*) and (*b*), respectively, illustrate the course of the deflection-repetition graph for annealed mild steel and for heat-treated chrome-nickel steel. As shown in Fig. 11 (*a*), the graph for water-cooled material is intermediate between the graphs for uncooled material tested at 50 and 1450 r.p.m. The temper colors as recorded in this figure indicate that the great decrease in deflection of the specimen tested at 1450 r.p.m. is due to its increase in strength at "blue heat." As shown in Fig. 11 (*b*), the uncooled specimen tested at 1450 r.p.m. increased its deflection more rapidly than did the other two specimens, thus indicating that for this material the strength decreases with increase in temperature. Fig. 11 should be compared with Figs. 8 and 9.

INFLUENCE OF HEAT INTERCHANGE BETWEEN SPECIMEN AND ENVIRONMENT

Effect of Thermal Conductivity of Environment.—By environment is meant the air, water or other medium surrounding the specimen. The term "thermal conductivity" will be used to signify specific rate of heat transfer whether by conduction, convection or radiation. The influence of heat interchange between specimen and environment on the upper portion of the stress-cycle graph has been considered in the discussion of cycle frequency and cyclic repetition. The influence of heat interchange on the portion of the stress-cycle graph, below the point of reversal of curvature, is well illustrated by Fig. 7, showing results obtained with annealed monel metal. Although material DYE had not been annealed after hot rolling, its tensile properties are those of fully annealed material.

As shown in Fig. 7, when the test specimen is surrounded by water at 65° F. (18.3° C.) instead of air at the same temperature, the stress-cycle graph is moved upward and to the right, and the curvature is considerably decreased. When ice water at 34° F. (1.11° C.) is used instead of water at 65° F. (18.3° C.), the position of the graph is practically the same as when water at 65° F. (18.3° C.) is used. When water at 185° F. (85° C.) is used, the graph is lowered slightly below but remains parallel to the graph for material tested in water at 65° F. (18.3° C.). Evidently, even when the initial temperature of the environment is raised about 120° F. (66.72° C.), the substitution of water for air moves the stress-cycle graph upward and to the right and decreases the curvature.

Rough temperature measurements on uncooled specimens were made by applying to a portion of the stressed surface a thin coating of samples of paraffin of different melting points. These measurements indicate that the surface temperature at the endurance limit does not exceed about 120 to 140° F. (49 to 60° C.). The higher position of the graph for material tested in water at 185° F. (85° C.) can be accounted for on the assumption that in the minute regions of maximum heat evolution the temperature is lower in specimens surrounded by water at 185° F. (85° C.), although the average surface temperature is higher than in the specimens surrounded by air. The lower temperature gradient, due to the greater conductivity of the environment, more than compensates for the higher initial temperature in the specimens tested in water at 185° F. (85° C.).

By substituting for air an environment of lower conductivity, the stress-cycle graph is moved downward and to the left. This is illustrated in Fig. 7 by results of tests on three specimens of material DY wrapped in asbestos wool.

The results illustrated in Fig. 7 would seem to indicate that the position of the horizontal asymptote of the stress-cycle graph varies with the conductivity of the environment. To confirm these indications experimentally would require tests to many more cycles than those illustrated in Fig. 7. Theoretical considerations, however, would lead to the same conclusion that is indicated by the results given in Fig. 7. Throughout a test at the endurance limit there is a definite course of change of the temperature gradient and of the temperature in the minute regions of maximum heat evolution. This implies a definite course of change in the rate of heat removal. Any change in conductivity of the environment, therefore, would alter the endurance limit.

The effect of conductivity of the environment on the form and position of the lower portion of the stress-cycle graph is of practical importance only when the hysteresis at the endurance limit, and the rate of increase of hysteresis with increase in stress, are relatively great. Such a condition is found in annealed or heat-treated steels and in annealed non-ferrous metals, but not in cold-worked metals. The effect of conductivity of the environment is greater the smaller the proportion of the heat conducted along the specimen to the holders. The proportion of the heat conducted to the holders is less the lower the thermal conductivity of the specimen and the greater the ratio of length and surface area to cross-section.

Effect of Thermal Conductivity of Specimen.—In the experiments that have just been described, the conditions were such that the influence of the thermal conductivity of the environment was great. The thermal conductivity of monel metal is about one-half that of steel, one-third that of bronze, one-fourth that of brass, one-eighth that of aluminum, and one-sixteenth that of copper. The low thermal conductivity of monel metal makes it possible, by varying the conductivity of the environment, to make proportionally large changes in the rate of total heat removal from the specimen. With increase in thermal conductivity of the specimen, other things being equal, the stress-cycle graph would be moved upward and to the right and the curvature of the lower portion would be decreased.

As shown in Fig. 7, the stress-cycle graph for annealed monel metal approaches the horizontal asymptote as abruptly and at as few cycles as does the graph for any steel. This is contrary to the wide-spread idea that the position of the stress-cycle graph for non-ferrous metals is always to the right of the graph for steels. It seems probable that the differences in form and position between the graphs for metals that are not cold-worked are due in part to the great differences in thermal conductivity. The high thermal conductivity of aluminum alloys and of some copper alloys is accompanied by a relatively distant position of the stress-cycle graph from the origin of abscissas, even in heat-treated or annealed alloys.

As shown in Fig. 8, varying the conductivity of the environment, or varying the rate of heat evolution by varying the cycle frequency, changes the form and position of the stress-cycle graph for steels as for monel metal. As the effect of water on steel, however, is not merely thermal, the stress-cycle graph for water-cooled steel specimens is not entirely comparable with that for monel metal.

Effect of Form and Type of Specimen.—Obviously, increasing the ratio of area of cross-section to length of specimen has qualitatively the same effect as increasing the thermal conductivity. Increasing the ratio of surface to length increases the relative importance of the conductivity of the environment. In a rotating-bend specimen the highly stressed region, being at the surface, is more accessible to the influence of conductivity of environment than in a direct-tension-compression specimen. The greater rate of heat evolution per unit area of cross-section, in the latter than in the former, however, would tend to establish a greater longitudinal temperature gradient. In a relatively long specimen of either type, especially if the thermal conductivity be low, the influence of thermal conductivity of the environment is at a maximum.

Effect of Cycle Frequency.—Decrease in the cycle frequency, as it decreases the rate of heat formation, would have the same effect as increasing the thermal conductivity of specimen and environment.

With decrease in cycle frequency, therefore, the lower portion of the stress-cycle graph is moved upward and to the right and the curvature is decreased.

Summary of the Effect of Heat Interchange Between Specimen and Environment.—The stress-cycle graph is moved upward and to the right and the curvature of the lower portion is decreased by the following variables: Increase in thermal conductivity of specimen or environment; increase in ratio of area of cross-section to length of specimen; and decrease in cycle frequency. The effect of these variables on the stress-cycle graph, therefore, is similar to the effect of cold working. There are evidently two opposing effects of cyclic deformation, just as there are two opposing effects of ordinary cold working. The one effect is a hardening of the metal, the other effect is a softening or "self-annealing" due to the heat evolved by the cold working. The hardening effect decreases, and the self-annealing effect increases, with increase in the degree of previous cold working. This is one reason why the endurance limit of annealed metals is above the elastic limit and the endurance limit of cold-worked metals is below the elastic limit. The hardening effect of cold working increases, and the "self-annealing" effect decreases, with increase in the rate of removal of heat or with decrease in the rate of deformation (cycle frequency). It is not intended to imply that "self-annealing" is the only influence opposing the hardening effect of cyclic deformation.

TABLE VI.—ORDINATE DIFFERENCES AND RATIOS "*b*" FOR ABSCISSA INTERVALS INDICATED.

Designation	Ordinate Difference 10^3–10^4 lb. per sq. in.	Ratio *b*	Ordinate Difference 10^4–10^5 lb. per sq. in.	Ratio *b*	Ordinate Difference 10^5–10^6 lb. per sq. in.	Ratio *b*	Ordinate Difference 10^6–10^7 lb. per sq. in.	Ratio *b*	Ordinate Difference 10^7–10^8 lb. per sq. in.	Extrapolated Ordinate Difference 10^8–10^9 lb. per sq. in.
DYD-8........	34 000	0.9	37 000	1.9	19 000	1.9	10 000	2.5	4 000	1 600
DYD-9........	27 000	0.8	36 000	1.9	19 000	1.7	11 000	2.7	4 000	1 500
DYC-8........	25 000	0.7	35 000	1.8	20 000	2.2	9 000	2.2	4 000	1 800
DYC-10.......	25 000	0.9	28 000	1.6	17 000	1.7	10 000	3.3	3 000	900
DYB-8........	26 000	0.8	31 000	1.7	19 000	1.9	10 000	2.5	4 000	1 600
DYA-8........	25 000	1.0	25 000	1.5	17 000	1.7	10 000	2.5	4 000	1 600
DYA-10.......	.....	...	20 000	1.2	16 000	1.6	10 000	3.3	3 000	900
DY...........	30 000	2.1	14 000	2.3	6 000	...	.....	...	....	....

EXTRAPOLATION OF STRESS-CYCLE GRAPHS

To save time in the practical determination of endurance limits the stress-cycle graph should be extended by experiment only far enough to permit of approximate determination of the asymptote by extrapolation. In order that extrapolation may be used it is necessary not only to know that the pure stress-cycle graph on a semi-logarithmic scale is a curve with horizontal asymptote, but also to know what kind of asymptotic curve it is. It has not yet been found possible to identify the stress-cycle graph as one of the well-known mathematical curves.

The curve may possibly be best defined as a modified exponential curve. In a simple exponential curve, the ordinate-differences for equal successive abscissa-intervals are in geometric proportion, and the total ordinates (measured from the horizontal asymptote) at these equal abscissa-intervals are in the same geometric proportion. The equation of an exponential curve may be written $y = \frac{a}{b^x}$ in which a and b are constants and b is the ratio of the above-mentioned geometric series.

In Table VI are listed for each curve in Fig. 1 successive ordinate differences and the ratios between these successive differences. These ratios have been designated b, although evidently the value of b is not constant throughout each curve as it would be throughout an exponential curve. For each of the first five materials listed in the table, the first value of b is less than 1.0. This represents the upward convexity in the upper part of the corresponding graphs in Fig. 1. Reading from left to right in the table the value of b then increases. In general, the increase in b is more rapid the softer the material.

The stress-cycle graph, therefore, approaches its horizontal asymptote more rapidly than if it were an exponential curve. If the graphs be extrapolated beyond 10^8 cycles, using the values for b listed in the column furthest to the right in the table, the values obtained for ordinate-differences will be somewhat larger than the true values. The values for the endurance limit thus obtained would be slightly on the safe side of the true value. The values for ordinate-differences in the last column of Table VI represent the extrapolated values for decrease in the ordinate between 10^8 and 10^9 cycles. Between 10^9 and 10^{10} cycles, extrapolated decreases would be negligible. Actually the decrease in ordinates between 10^8 and 10^9 cycles is practically negligible.

It should now be possible to obtain by extrapolation the approximate endurance limit of any cold-worked non-ferrous metal, if the position of the stress-cycle graph between 10^5 and 10^7 cycles be known. By using the value of b obtained from the graph, the decreases in the ordinate between 10^7 and 10^9 cycles could be estimated with sufficient accuracy.

If the graph cannot readily be extended backward to 10^5 cycles, it is sometimes necessary to extend it outward by experiment to about

50,000,000 cycles before extrapolation can be done with sufficient accuracy. The stress-cycle graph for duralumin given in a previous paper[1] is a good example of the method as applied to such a graph. The ordinate of the duralumin graph decreases about 8000 and 3000 lb. per sq. in., respectively, in the abscissa intervals 10^6 to 10^7 and 10^7 to 10^8. If the same ratio holds, the ordinate would decrease about 1000 lb. per sq. in. in the interval 10^8 to 10^9. Since the ratio probably increases, however, the actual decrease of ordinate in the latter interval is probably not more than about 500 lb. per sq. in.

PART III.—CORROSION-FATIGUE OF METALS

ORIGIN OF INVESTIGATION

The investigation of the effect of a water stream on the properties of monel metal under endurance test led to information of such value that it seemed desirable to extend the investigation to steel. Investigation of the upper part of the stress-cycle graph for steel by means of water-cooled specimens gave results similar to those obtained with monel metal, except for steel that is stronger at "blue heat" than at room temperature. Attempt to extend the investigation to the lower part of the graph, however, gave apparently abnormal results which seemed to call for further study. Additional experiments made for this purpose showed that the abnormality is due not to the thermal but to the chemical effect of water on steel, and that this chemical action has unexpected influence on the "fatigue-resisting" properties of the steel.

STRESS-CYCLE GRAPH FOR SPECIMENS TESTED IN WATER STREAM TO FAILURE

Results obtained with specimens tested in a water stream to failure are indicated in Figs. 8 and 9 by small open triangles and the broken line passing through them. The first experiments (made with 5-per-cent nickel steel, Material CB-W-10 as illustrated in Fig. 8) showed that the stress-cycle graph for water-cooled material at about 100,000 cycles begins to descend rapidly below the graph for material tested in air. This graph was followed down to a stress of 40,000 lb. per sq. in., at which it showed the first signs of approach to a horizontal asymptote.

When this effect was observed, the investigation was extended to chrome-nickel steel and carbon steel in various conditions of heat treatment, ingot iron, and stainless iron. As shown in Figs. 8 and 9 the graphs for material tested in a water stream to failure all descend below the graphs for material tested in air. These graphs also indicate that the damaging effect of the water stream on the fatigue-resisting properties of the steel increases with increase in hardness. The effect on ingot iron within the range investigated is slight. Stainless iron under these conditions is not so much superior to ordinary steels as had been expected.

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[1] D. J. McAdam, Jr., "Endurance Properties of Non-Ferrous Metals, Part II," Paper 1537D, Am. Inst. Mining and Metallurgical Engrs. Issued with Mining and Metallurgy, February, 1926.

CAUSE OF THE DAMAGING EFFECT OF A WATER STREAM ON THE ENDURANCE PROPERTIES OF STEEL

It will be noticed that for the harder steels the most rapid descent of the stress-cycle graph for water-cooled specimens below the graph for specimens tested in air is between abscissas of 100,000 and 300,000 cycles. The range of time for such a cycle range is between about $1\frac{1}{4}$ and 4 hours. As the specimens so tested were entirely surrounded by the water stream they had not become rusty at the end of this time interval but appeared fairly bright. On closer examination, however, small spots of dull appearance were visible. After 100,000 cycles in the water the spots were hardly visible without a hand lens. When these spots were examined at higher magnification the cause of the damage to the endurance properties was apparent.

The appearance of these spots is shown at a magnification of 100 in Fig. 12. As shown in Figs. 12 (*a*) and 12 (*f*) some of these spots are oxide patches, each surrounding a non-metallic inclusion or pit from which an inclusion has been removed. As shown in Figs. 12 (*b*), (*c*), (*d*) and (*e*), some of the spots show no decided oxide patches, but consist of inclusions (or pits from which inclusions have been removed) from which cracks have started. The cracks are all nearly transverse to the axis of the specimen. The absence of oxide patches from many of the spots is undoubtedly due in part to the fact that specimens on removal from the machine were rubbed with soft cotton and oil before being prepared for photographing, and in part to erosion by the water stream. What appear to be longitudinal scratches are chiefly due to the longitudinal rubbing of the oxidized surface with oiled cotton wool.

Examination of specimens that had been tested to failure in air showed no such spots and cracks.

The evidence presented by these photomicrographs would indicate that in these failed specimens tested in a water stream numerous short transverse cracks have formed, and that one of these has advanced so far as to cause failure of the specimen. Evidently, therefore, the damage to the endurance properties of such specimens is due to the combined action of corrosion and fatigue, localized at regions where corrosion and stress are the greatest. This combined action has started transverse cracks, which are regions of such stress concentration as to cause failure at nominal stresses below the endurance limit. Such failure under combined corrosion and fatigue may be called "corrosion-fatigue."

EFFECT OF VARYING DEGREE OF CORROSION

As the effect of combined corrosion and fatigue was found to be so great, it became necessary to investigate the effect of varying degrees of corrosion, and to determine whether or not for each degree of corrosion there is a fairly definite fatigue limit. For this purpose several series of experiments were made in which the water stream was stopped at different intervals after the first application of stress. After the water stream was stopped the specimen was rubbed gently with oiled cotton wool and the fatigue test was continued until the specimen failed. The results of these experiments are illustrated in Figs. 8 and 9. As the stainless iron was from four heats of steel that had been received at various times on the same specification, it was not considered sufficiently uniform to permit investigation of the effect of varying degree of corrosion. Ingot iron was not available in sufficient quantity to permit such an investigation. For these materials, however, results obtained with specimens tested in a water stream until failure are illustrated in Fig. 9. The zig-zag lines in some of the stress-cycle graphs indicate that the specimen was tested at the stress and for the number of cycles indicated by the lower end of the line, and that the stress was then raised at intervals as indicated by the course of the line.

Fatigue Tests with Specimen in Water Stream for First 300,000 Cycles.—The results of these experiments are illustrated in Figs. 8 and 9 by the curves marked "A." It will be noticed that these curves are practically continuations of the curves representing specimens water-cooled throughout the entire test. The curves "A" evidently become nearly horizontal between one and ten million cycles. A comparison of the results for these steels indicates that the effect of corrosion on fatigue-resistance increases with increase in the hardness of the steel. This is well illustrated by comparing in Fig. 8 curves "A" for materials BD-W-10, BD-W-11, BD-W-12 and BD-15, which represent the same chrome-nickel steel heat-treated in various ways. A comparison in Fig. 9 of curves "A" for materials AG-W-9 and AG-15.5 leads to the same conclusion. The "corrosion-fatigue" limits for this degree of corrosion have been designated S_{c_1} and listed

TABLE VII.—COMPARISON OF CORROSION-FATIGUE LIMITS WITH ENDURANCE LIMITS AND WITH OTHER PHYSICAL PROPERTIES.

Material	Designation	Tensile Strength, lb. per sq. in.	Total Elongation in 2 in., per cent	Elongation in 2 in. at Maximum Load, per cent	Reduction of Area, per cent	Charpy Value, ft-lb.	Endurance Limit, lb. per sq. in. *Sf*	Corrosion-Fatigue Limits, lb. per sq. in.					Ratios				
								Sc_1	Sc_3	Sc_{15}	*Sct*	*Spc*	Sc_1 to *Sf*	Sc_3 to *Sf*	Sc_{15} to *Sf*	*Sct* to *Sf*	*Spc* to *Sf*
5-per-cent Nickel Steel	CB-W-10	129 600	21.8	9.6	56.4	28.0	61 000	......	35 000	27 000	......	......	....	0.57	0.44	....	....
Chrome-Nickel Steel	BD-W-10	148 400	18.2	4.2	54.0	27.6	73 000	51 000	43 000	29 000	......	66 000	0.70	0.59	0.40	....	0.90
Chrome-Nickel Steel	BD-W-11	132 900	21.9	6.5	57.0	25.2	67 000	48 000	38 000	29 000	......	61 000	0.72	0.57	0.43	....	0.91
Chrome-Nickel Steel	BD-W-12	115 800	25.6	9.5	61.3	40.6	58 000	47 000	37 000	27 000	......	52 000	0.81	0.64	0.47	....	0.90
Chrome-Nickel Steel	BD-15	105 800	26.2	15.0	52.2	24.5	51 000	41 000	35 000	28 000	......	45 000	0.80	0.69	0.55	....	0.88
0.36-per-cent Carbon Steel	AG-W-9	103 600	25.0	11.0	57.8	37.8	51 000	41 000	34 000	26 000	......	45 000	0.80	0.67	0.51	....	0.88
0.36-per-cent Carbon Steel	AG-15.5	79 200	31.3	18.8	48.7	22.4	34 000	33 000	29 000	27 000	......	33 500	0.97	0.85	0.79	....	0.99
Stainless Iron	ZZ	107 300	21.3	6.3	67.5	48.6	59 500	......	......	......	34 000	......	....	....	....	0.57	....

Sf Endurance limit.
Sc_1 Corrosion-fatigue limit; water stream 100 000 cycles.
Sc_3 Corrosion-fatigue limit; water stream 300 000 cycles.
Sc_{15} Corrosion-fatigue limit; water stream 1 500 000 cycles.
Sct Corrosion-fatigue limit; water stream until failure.
Spc Corrosion-fatigue limit; corrosion previous to stress.

in Table VII. It will be noted that with this degree of corrosion the advantage of heat treatment in conferring fatigue resistance has been greatly reduced but not entirely eliminated.

Fatigue Tests with Specimen in Water Stream for First 1,500,000 Cycles.—In order to determine the effect of more severe corrosion, a series of fatigue tests was then made with the water stream in contact with the specimen for the first 1,500,000 cycles (about 18 hours). When the water stream was stopped, the specimens were found to be definitely rusted, but most of the rust was readily removed by the above-described method of rubbing and oiling. Numerous small spots, often of rusty appearance, remained. These spots in specimens tested above the corrosion-fatigue limit were oxide patches containing transverse cracks as shown in Fig. 12 (*f*). The results of these experiments are illustrated in Figs. 8 and 9 by the curves marked "B." For all but the softer steels there is apparently a reversal of curvature at the departure of curve "B" from the curve for specimens tested in water stream until failure. This reversal is probably due to the removal at this point of the cooling effect of the water from the locally heated microscopic cracks. Curve "B," however, evidently approaches a horizontal asymptote which would depend not on the preliminary cooling, but on corrosion-fatigue alone.

It will be noted that the corrosion-fatigue limit for this degree of corrosion differs very little for all the steels tested, except for ingot iron. Under these conditions the advantage of heat treatment in conferring fatigue resistance is practically eliminated.

Fatigue Tests with Specimen in Water Stream for First 100,000 Cycles.—It seemed desirable to investigate also the effect of a very slight degree of corrosion. For this purpose experiments were made with specimens in water stream for the first 100,000 cycles (about $1\frac{1}{4}$ hours). As stated above, the degree of corrosion thus produced is hardly noticeable to the unaided eye. Results of these experiments are illustrated in Figs. 8 and 9 by the curves marked "C." These curves show that the amount of corrosion need be very slight to damage the fatigue-resistance of steel, especially quenched-and-tempered steel.

The relative position of curve "C" varies with the hardness of the steel. For materials BD-W-10 and BD-W-11, for example, curve "C" is nearer to curve "B" than to the curve "E" for uncorroded specimens. For materials BD-W-12 and BD-15, curve "C" is nearer to curve "E" than to curve "B." For this slight degree of corrosion the advantage of heat treatment in conferring fatigue-resistance is considerably reduced.

Minimum Degree of Corrosion Necessary for Appreciable Damage to Fatigue Resistance.—It is of interest to know the minimum degree of corrosion that will damage appreciably the fatigue resistance of steel. This subject has not yet been thoroughly investigated. A single specimen of material CB-W-10 was tested in a water stream for the first 50,000 cycles, then in air until failure. This specimen gave no evidence of corrosion-fatigue. Its endurance properties were those of uncorroded steel. Evidently, therefore, for this material the minimum degree of corrosion necessary to damage the fatigue-resistance is produced by contact with a water stream for 50,000 to 100,000 cycles. The limiting degree of corrosion, as measured by the number of cylces in a water stream, would vary with the corrosion resistance of the material. This suggests a possible method of comparing corrosion resistance for steels.

Effect of Corrosion Prior to Fatigue.—The above-described effects were produced by simultaneous corrosion and fatigue. It now seemed desirable to determine whether corrosion and fatigue, if not simultaneous, would produce similar effects. For this reason experiments were made in which corrosion was prior to fatigue. In one series, the specimen without stress was subjected to a water stream for 1,500,000 revolutions. A few specimens that had been tested in air at stresses below their endurance limits were then utilized to determine the effect of intermittent dipping in normal salt solution for 17 to 24 hours prior to fatigue test. One specimen was then merely dried in the air. The others were wiped with oiled cotton wool. These specimens were then subjected to fatigue at various stresses.

The effect of prior corrosion is about the same for either of the methods described above. A comparison of curves "D" and "C" shows that the effect of rather severe corrosion prior to fatigue is much less than the effect of very slight corrosion simultaneous with fatigue. Even this relatively small effect of severe prior corrosion is probably chiefly due to slight corrosion under the rust patches continuing simultaneously with the fatigue. In corrosion-fatigue, as in "season-cracking," corrosion and stress are evidently simultaneous.

RELATION OF CORROSION-FATIGUE LIMITS TO THE ENDURANCE LIMIT AND TO OTHER PHYSICAL PROPERTIES

Corrosion-fatigue limits and endurance limits taken from Figs. 8 and 9 are listed in Table VII. Ratios between these corrosion-fatigue limits and endurance limits are also listed in this table. The ratios of S_{c1} to S_f vary from 0.97 to 0.70 with increase in the hardness of the steel. The ratios of S_{c3} to S_f vary from 0.85 to 0.57. The ratios of S_{c15} to S_f vary from 0.79 to 0.40. The ratios of S_{pc} to S_f vary from about 0.99 to 0.88.

Apparently the "elongation at maximum load" is the best index of the ratio of corrosion-fatigue limit to endurance limit. The total elongation is the next best index. Charpy values show no definite relationship to these ratios. Steel having a high combination of tensile strength and elongation at maximum load, therefore, would apparently be best adapted for resistance to corrosion-fatigue.

Practical Importance of Corrosion-Fatigue

That the facts presented are of great practical importance is evident. It would be difficult to avoid in machinery the slight degree of corrosion that is sufficient to make a "corrosion-fatigue limit" rather than the "endurance limit" the most important physical property. Even slight intermittent corrosion, hardly sufficient to be noticeable in service, would probably be sufficient to reduce the corrosion-fatigue limit to values such as those designated in Table VII as S_{c15}. Under such conditions the advantage of heat treatment of steel would be practically eliminated. It seems probable that many mysterious failures of steel at stresses far below the endurance limit have been due to such corrosion-fatigue.

Corrosion-fatigue is undoubtedly of more importance in machinery parts subjected to repeated bend or torsion than in parts subjected to repeated axial stress. Corrosion-fatigue is of more importance the smaller and thinner the section of metal, but the variation of corrosion-fatigue with section is yet to be investigated.

It seems probable that corrosion-fatigue is of importance for non-ferrous metals. The great ductility of some non-ferrous metals, however, may make the ratio of corrosion-fatigue limit to endurance limit large. Moreover, for some conditions of service it is possible that the endurance limit of a non-ferrous metal and the corrosion-fatigue limit of steel might be the competing properties. The higher endurance ratio of steel might thus be offset by a higher ratio of corrosion-fatigue limit to endurance limit or by the absence of corrosion-fatigue for non-ferrous metals. Evidently, however, a large field has been opened up for investigation.

Acknowledgements

Acknowledgement is hereby made of the encouragement received in this work from Captain P. B. Dungan, U.S.N., Officer in Charge, U. S. Naval Experiment Station, and of the assistance received from Mr. G. F. Wohlgemuth, Associate Metallurgist, and from Messrs. John L. Basil and A. P. Vandermast, Laboratorians, and from John K. Amoss, toolmaker, at the Naval Experiment Station. The generous cooperation of the International Nickel Co. and the American Rolling Mill Co. is also acknowledged.

[For Discussion on Fatigue of Metals, see page 269.—Ed.]

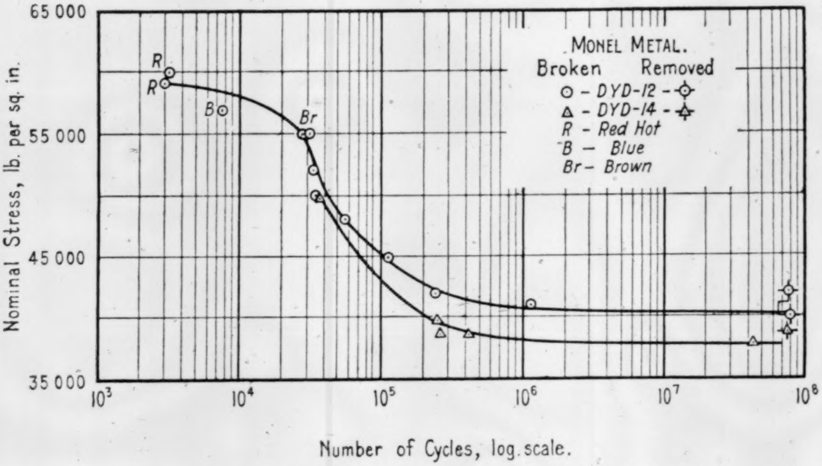

Fig. 2.—Stress-Cycle Graphs for Annealed Monel Metal.

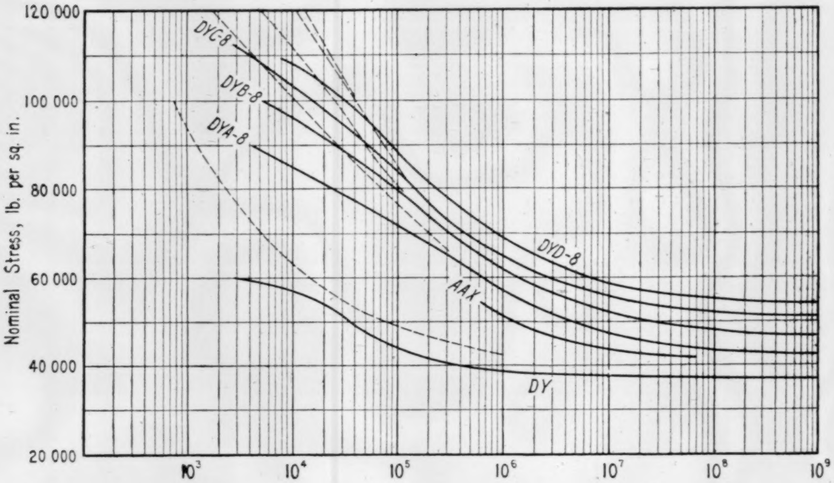

Fig. 3.—Effect of Cold-Working on Form and Position of the Stress-Cycle Graph for Monel Metal.

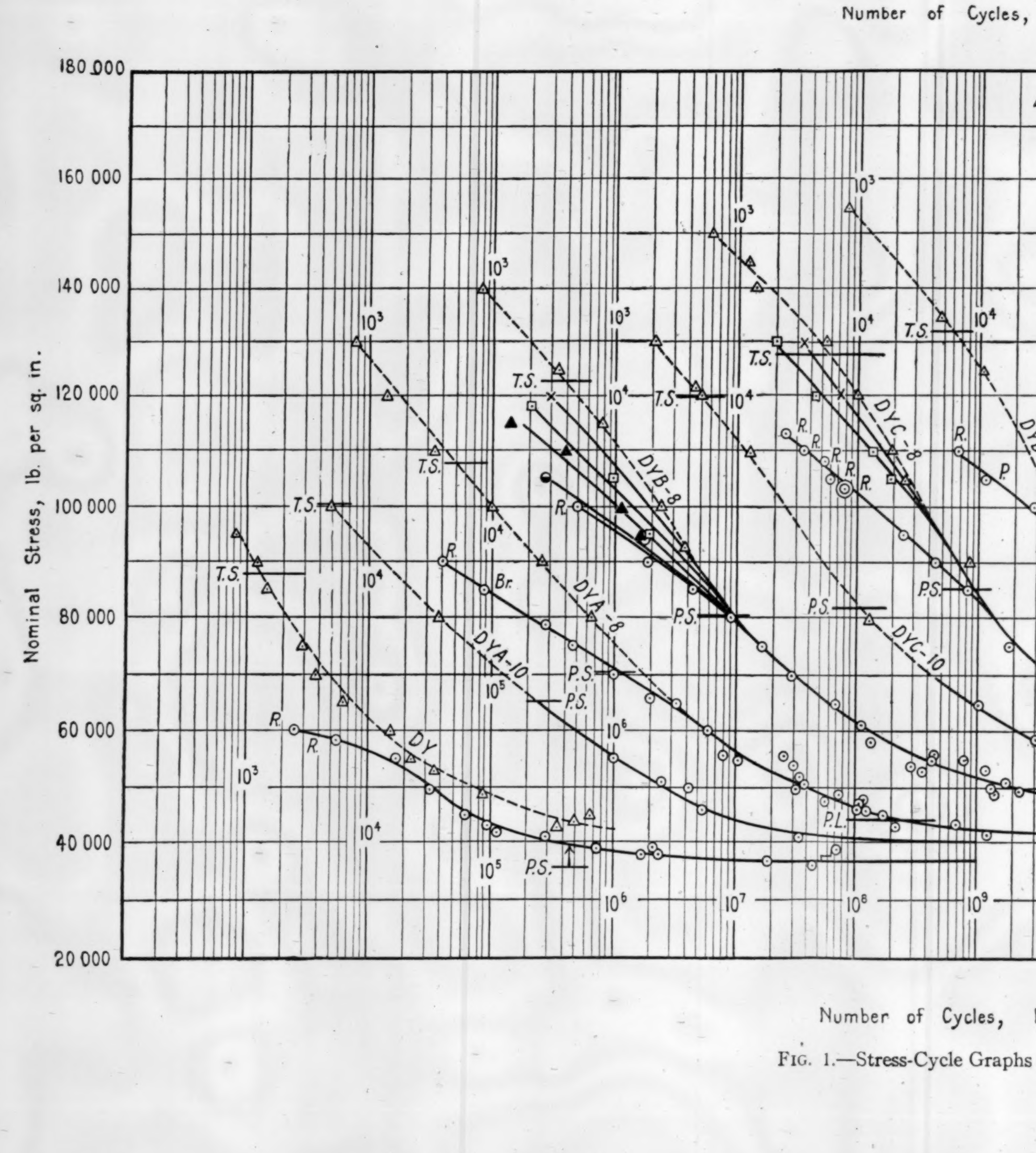

FIG. 1.—Stress-Cycle Graphs

PLATE I.
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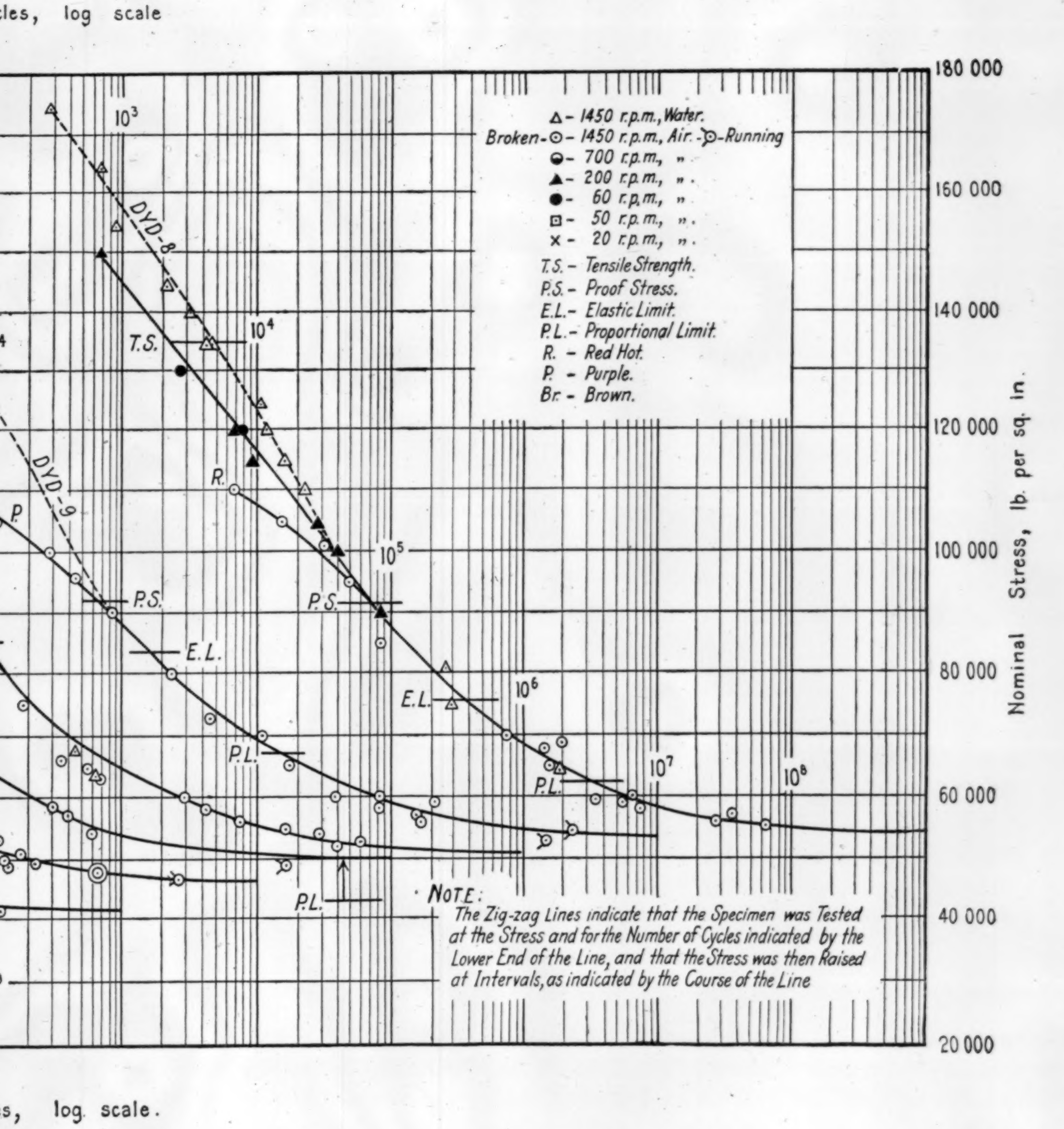
MCADAM ON CORROSION-FATIGUE OF METALS



raphs for Monel Metal.

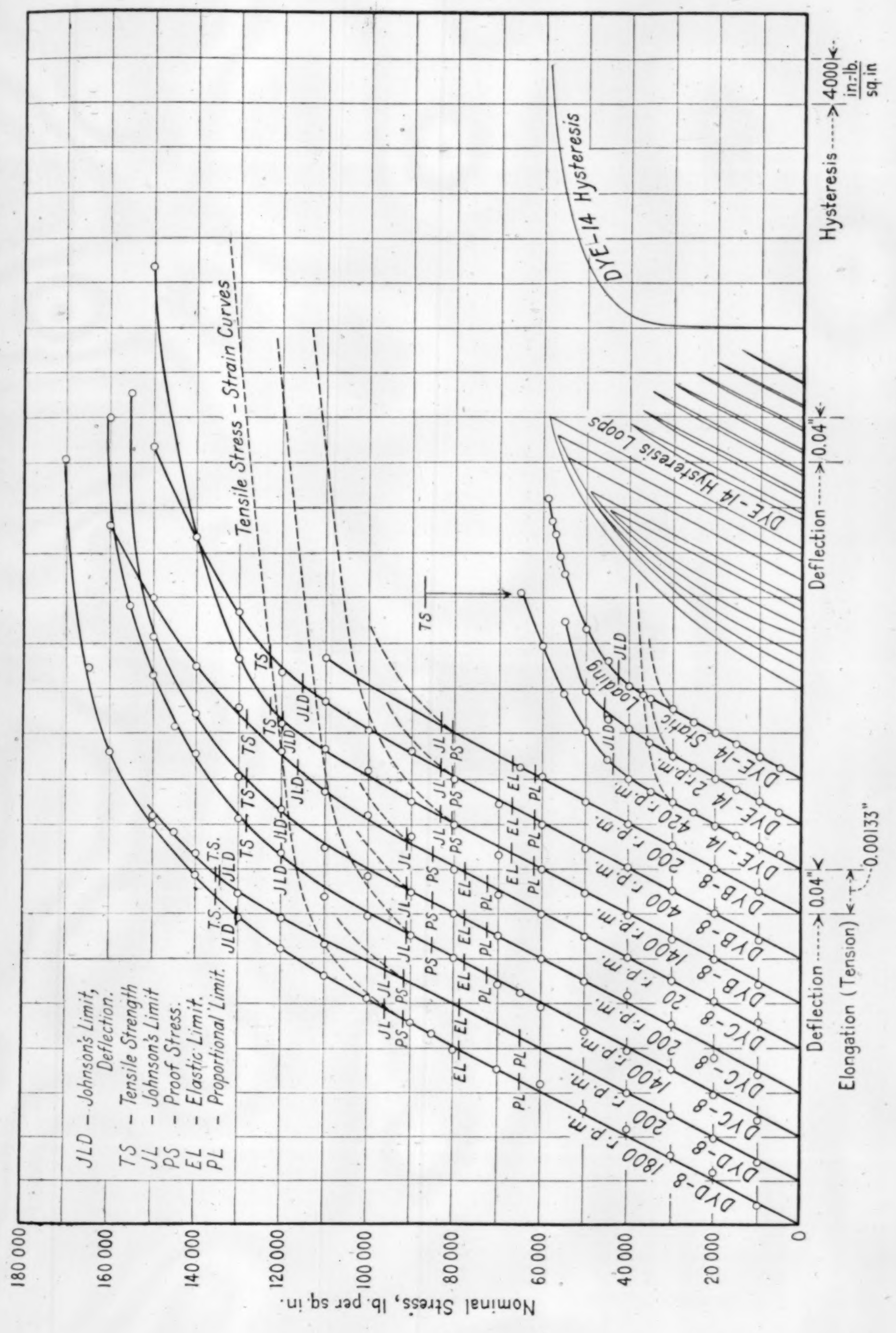


FIG. 4.—Stress-Deflection and Hysteresis Graphs for Monel Metal.

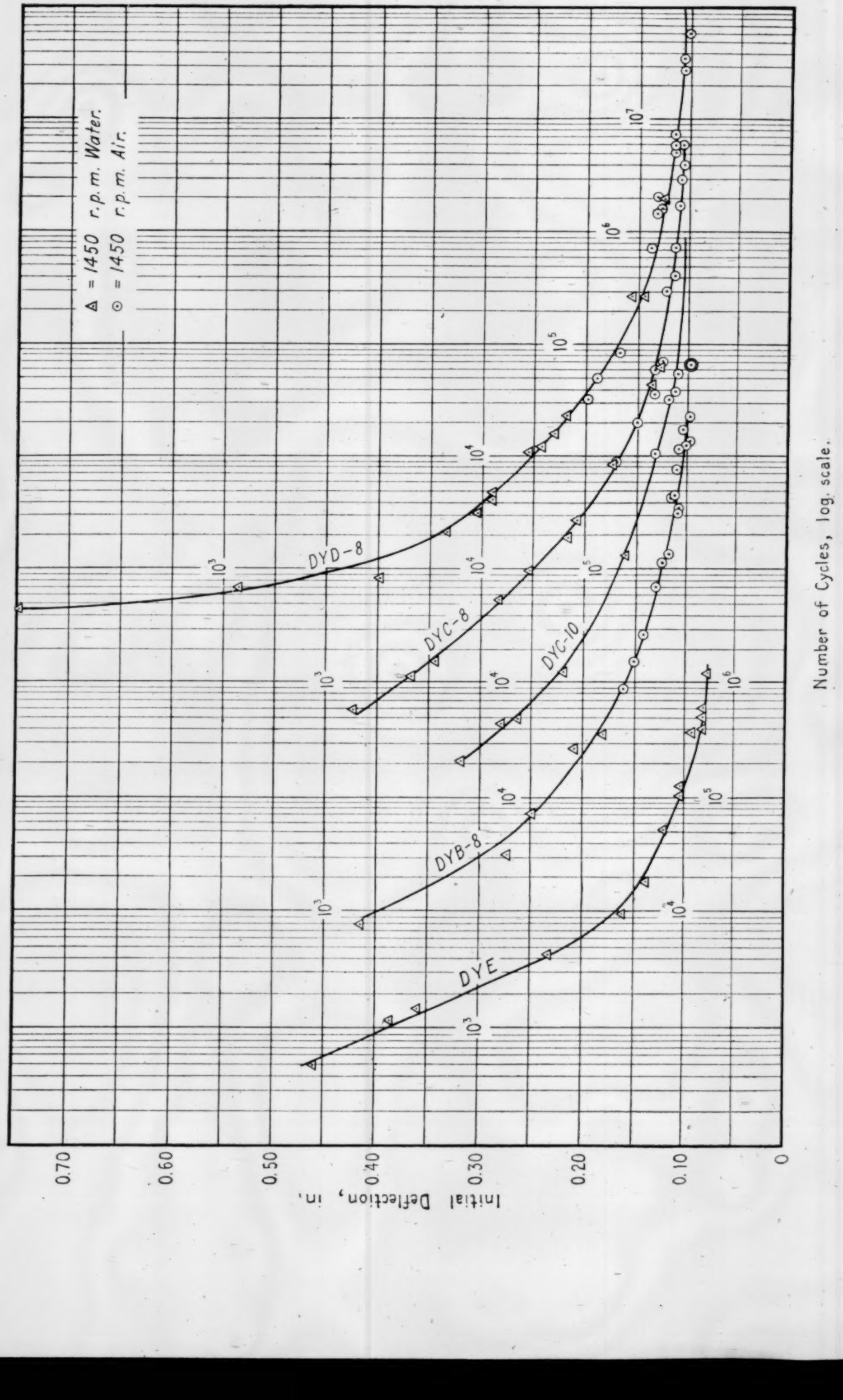


FIG. 5.—The Deflection-Cycle Graph for Monel Metal.

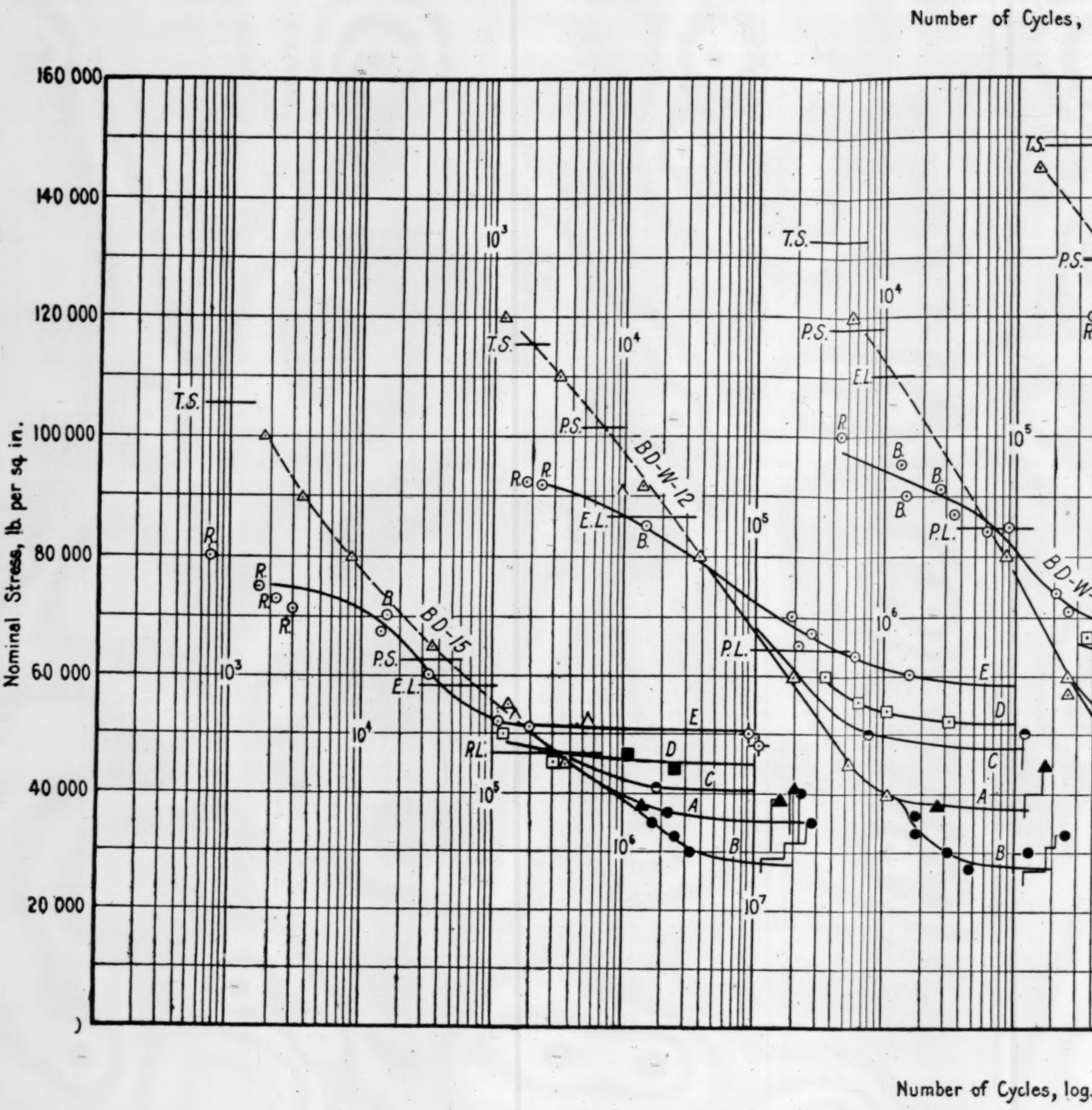


Fig. 8.—Corrosion-Fatigue o

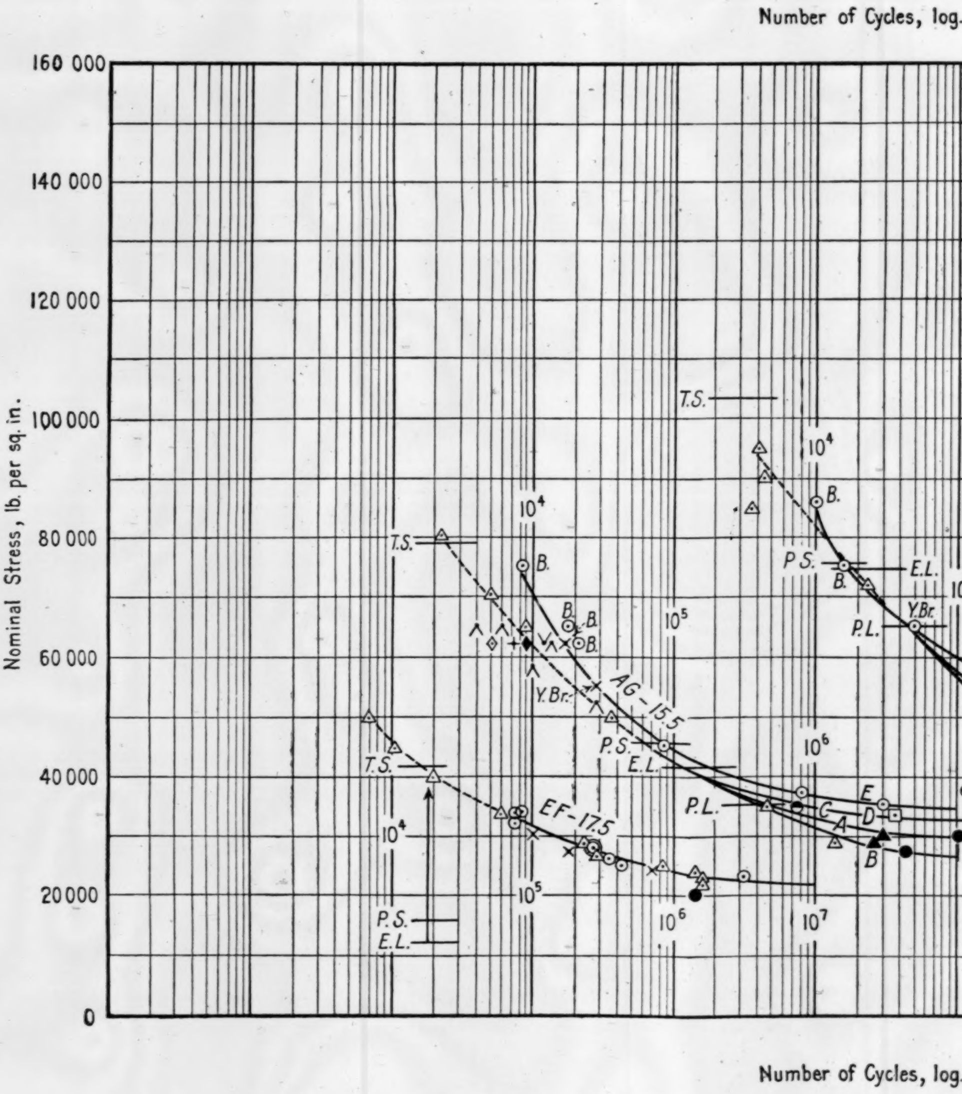
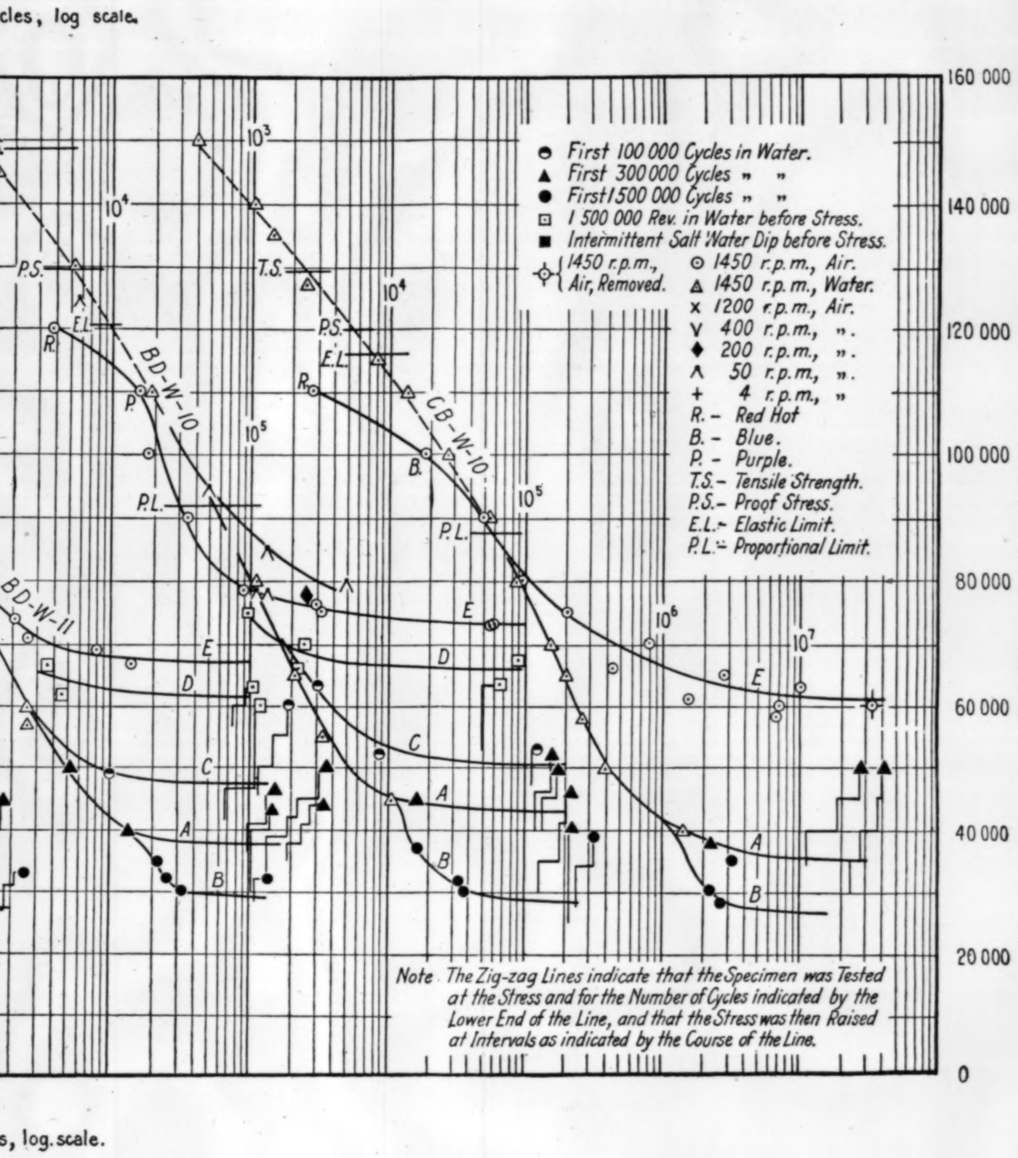
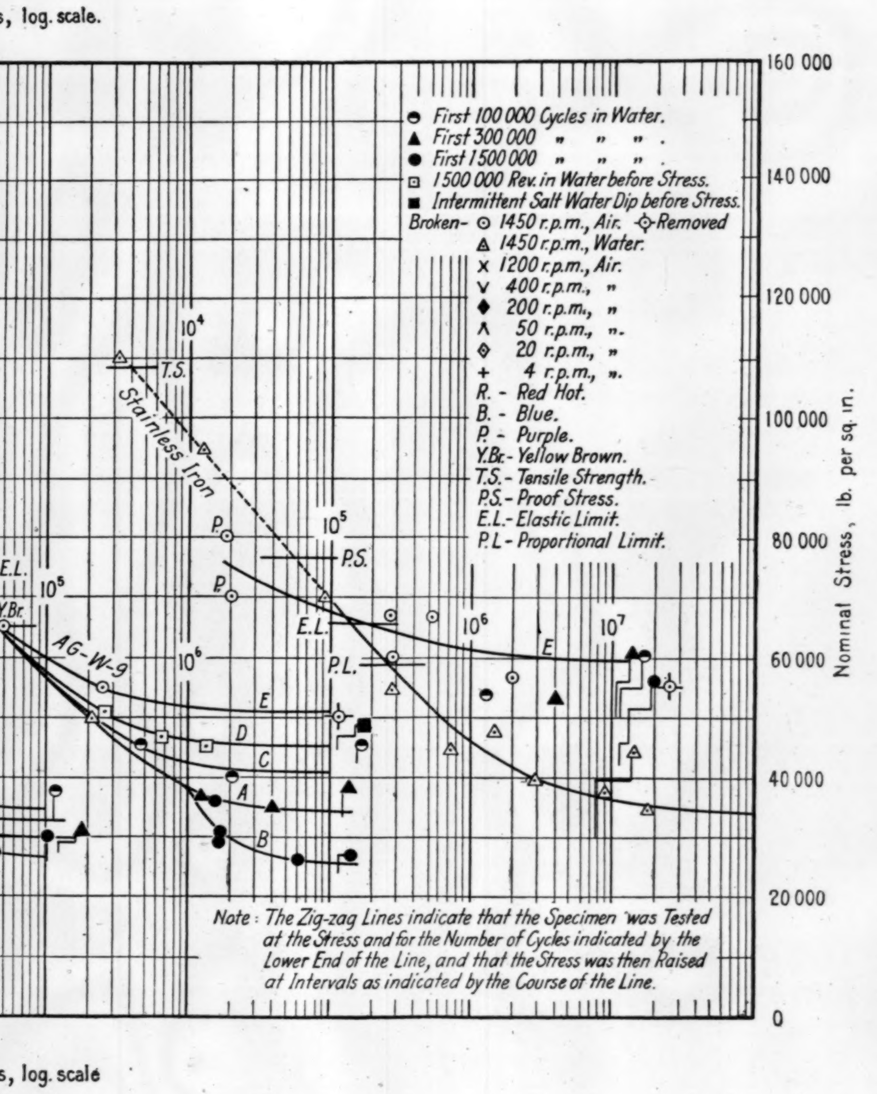


Fig. 9.—Corrosion-Fatigue of Carbon St



Note The Zig-zag Lines indicate that the Specimen was Tested at the Stress and for the Number of Cycles indicated by the Lower End of the Line, and that the Stress was then Raised at Intervals as indicated by the Course of the Line.

gue of Alloy Steels.



Note: The Zig-zag Lines indicate that the Specimen was Tested at the Stress and for the Number of Cycles indicated by the Lower End of the Line, and that the Stress was then Raised at Intervals as indicated by the Course of the Line.

on Steels and "Stainless Iron."

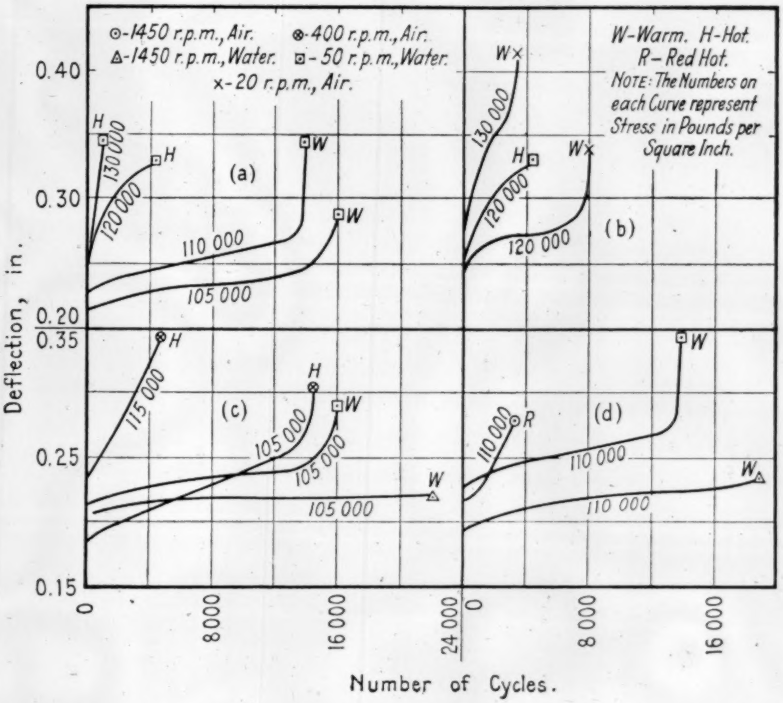


FIG. 6.—Effect of Cyclic Repetition on Deflection, Monel Metal, Material DYC-8.

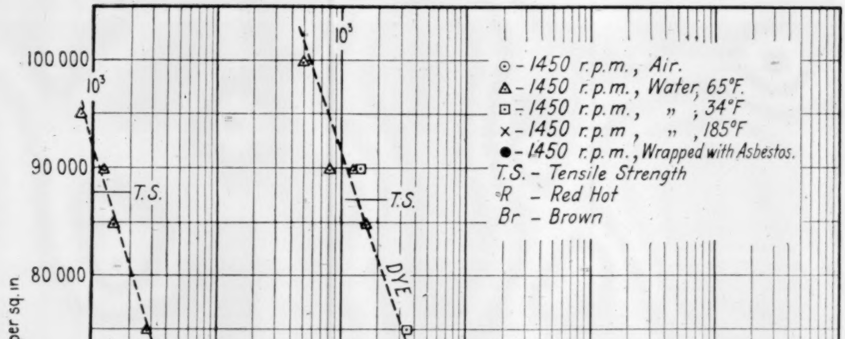


Fig. 7.—Effect of Heat Interchange Between Specimen and Environment.

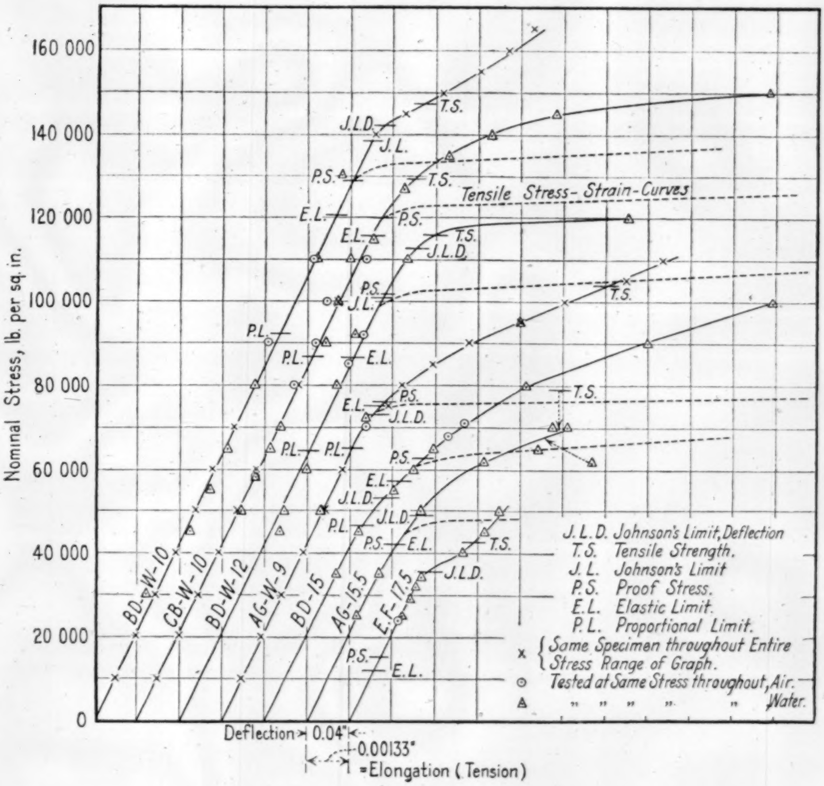


FIG. 10.—The Stress-Deflection Graph for Steel.

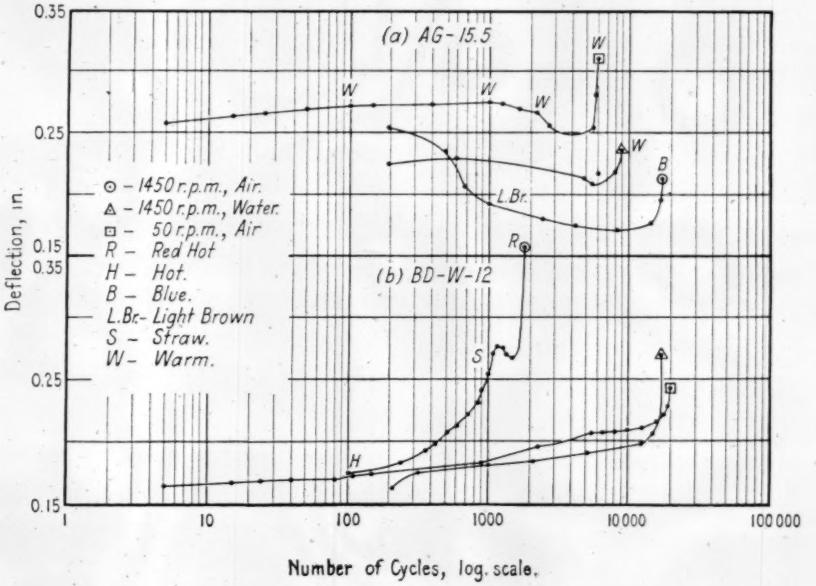
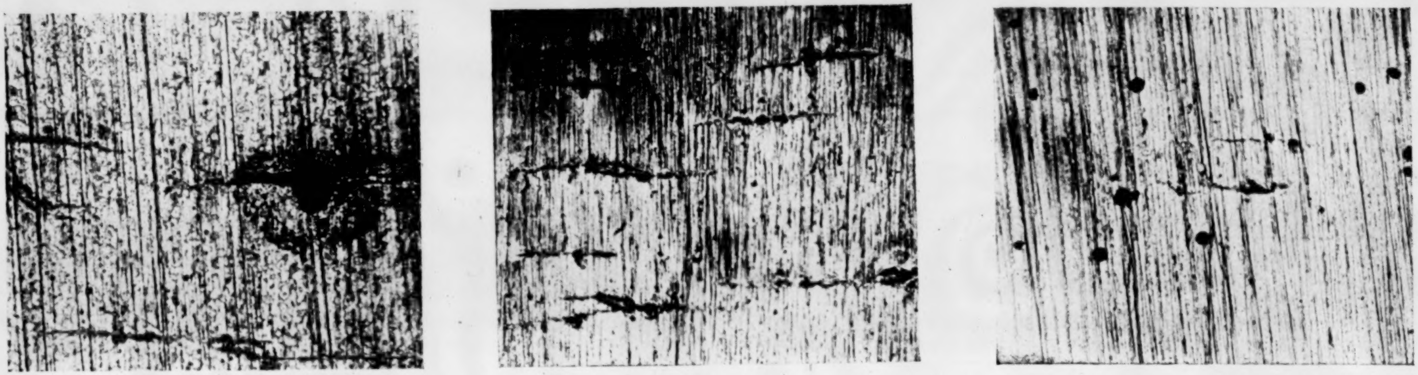


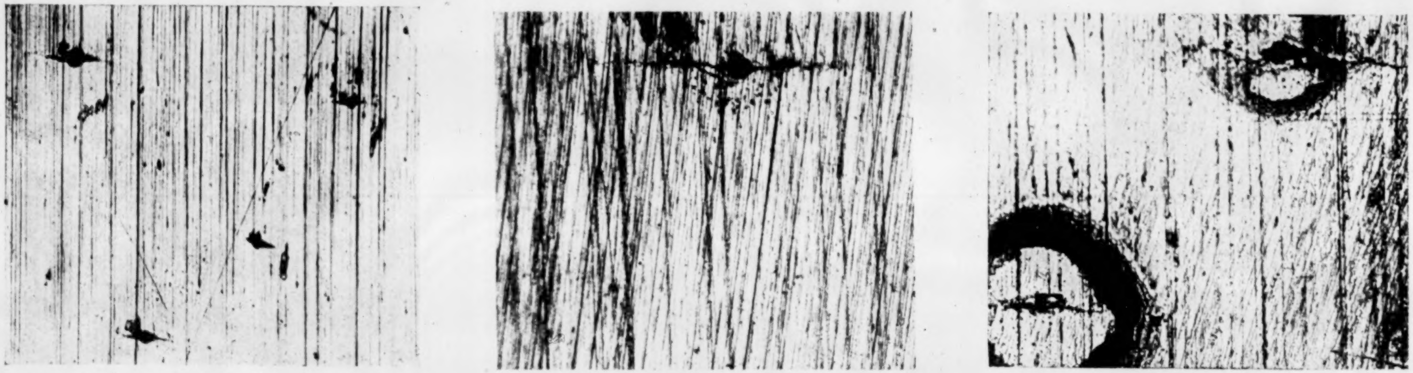
FIG. 11.—Effect of Cyclic Repetition on Deflection of Steels, Materials AG-15.5 and B D-W-12.



(*a*) 5-per-cent Nickel Steel, Material CB-W-10; Stress, 40,000 lb. per. sq. in.; in Water Throughout Test; 1,471,000 Cycles.

(*b*) Chrome-Nickel Steel, Material BD-W-10; Stress, 45,000 lb. per sq. in.; in Water Throughout Test; 1,131,000 Cycles.

(*c*) Stainless Iron, Material Z Z; Stress, 35,000 lb. per sq. in.; in Water Throughout Test; 18,404,500 Cycles.



(*d*) Chrome-Nickel Steel, Material BD-15; Stress, 41,000 lb. per sq. in.; in Stream of Water for 100,000 Cycles; Failed at 1,922,900 Cycles.

(*e*) 0.36-per-cent Carbon Steel, Material AG-W-9; Stress, 37,000 lb. per sq. in.; in Stream of Water for 300,000 Cycles; Failed at 1,262,600 Cycles.

(*f*) 5-per-cent Nickel Steel, Material CB-W-10; Stress, 35,000 lb. per sq. in.; in Stream of Water for 1,444,000 Cycles; Failed at 3,362,000 Cycles.

Fig. 12.—Photomicrographs of Corrosion-Fatigue Specimens (× 100).

EFFECT OF GROOVES, THREADS, AND CORROSION UPON THE FATIGUE OF METALS

By R. R. Moore[1]

Synopsis

It has been known for a long time that sudden changes in section such as sharp shoulders, holes, grooves, threads, etc., produce intensified local stresses, the presence of which is considered dangerous. This paper demonstrates the relative effect of this factor on the static tension properties and the resistance to repeated stresses (fatigue), also the effect of size of radius at root of groove and depth of groove. Further tests show the relative effects of a continuous thread and a radial groove of the same form. Tests on corroded steel show the effects of corrosive action.

Test results demonstrate that the static tensile strength is generally increased by the presence of the groove. While this has been known for some time, these particular tests show a direct comparison with the weakening effect of the notch when subjected to repeated stresses. Curves are given to show the increase in endurance limit with increase in radius at the bottom of the groove and the decrease with increase in depth of groove. Specimens which contain a continuous thread show much greater (2½ to 3 times) resistance to repeated stresses than specimens with a groove of the same form. Some brief corrosion tests show a reduction in endurance limit of about 21 per cent.

Comparisons of the effects of various factors such as chemical composition, heat treatment, mechanical treatment, corrosion, methods of joining (welding, brazing, soldering), grooves, notches, various sudden changes in section, etc., upon the applicability of metals as structural materials has been mainly based upon the results of the static tension test. It should be observed, however, that the static tension test is far from being representative of the actual service conditions of members of machines or structures. In the static tension test the metal fails after a single application of a gradually increasing load. The failures found in members of machines seldom, if ever, occur under the application of a single load. Such service failures are closely associated with the phenomena involved in failure under the repeated application of stresses. Such failures are progressive, due to the gradual spreading and growing of very fine cracks, with practically no deformation of the metal. This is an entirely different phenomenon than occurs in the static tension test. It is reasonable to expect, and experiment demonstrates, that the two tests are affected differently by different factors.

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[1] Chief of Physical Testing Branch, Engineering Division, U. S. Air Service, McCook Field, Dayton, Ohio.

It is the particular purpose of this paper to emphasize the fact that the usual interpretations of the results of the static tension test are of little use as a basis for design or selection of materials when the members are to be subjected to repeated stresses or vibration. In fact the tension test may prove very misleading in contradicting the real effect of service conditions as will be shown in the case of the effects of at least one factor: namely, grooves or threads.

The detrimental effect of sudden changes in section, such as sharp shoulders, grooves, notches, holes, threads, etc., upon the service life of machine members is evidenced by the large number of failures occurring at these localities. The tests described in this paper were prompted by service failures in airplane propeller hubs at the root of the thread on the hub used to hold the blade, and also in the threaded ends of steel tie rods which are subjected to considerable vibration.

TABLE I.—CHEMICAL COMPOSITION OF MATERIALS USED IN INVESTIGATION

Metal	Chemical Composition								
	Carbon, per cent	Phosphorus, per cent	Sulfur, per cent	Manganese, per cent	Chromium, per cent	Vanadium, per cent	Copper, per cent	Silicon, per cent	Iron, per cent
6130	0.29	Less than 0.040	0.021	0.77	1.07	0.18	....	....	....
25S	....	....	....	0.71	....	....	4.15	0.76	0.32
Tie Rod	0.43	Less than 0.040	0.040	0.60	....	....	....	....	....

This paper describes three series of tests as follows:

1. Tests on heat-treated 6130 steel, and the aluminum alloy 25S to determine the effect of shape of groove, depth of groove, and radius at bottom of groove.
2. Tests on tie rod steel to compare the relative effects of continuous threads and radial grooves of the same form.
3. Tests on steel to determine the general effects of corrosion (rusting).

MATERIALS

Airplane propellers are being made from both steel and light alloys. Test specimens were prepared from both metals. The steel specimens were cut from 1-in. diameter forged bars of 6130 steel of the composition given in Table I. Test specimens were heat treated as follows: Quenched in oil from 1675° F. (915° C.) and drawn at 1200° F. (650° C.). The light alloy specimens were cut from $5\frac{1}{2}$-in. square forged bars of 25S alloy of composition given in Table I.

The specimens from tie rod steel for comparing threads and radial grooves were taken from the round ends of streamline wire tie rods.

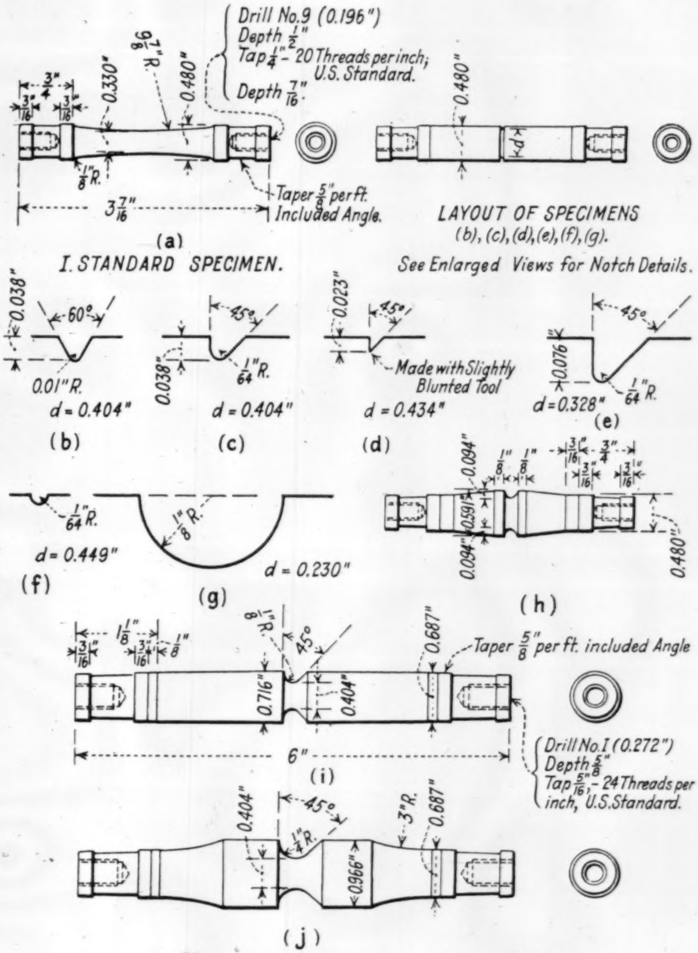

Drill No. 9 (0.196")
Depth 1/2"
Tap 1/4"-20 Threads per inch, U.S. Standard.
Depth 7/16"
9 7/8" R.
0.330"
0.480"
3/4"
3/16"
3/16"
1/8" R.
Taper 5/8" per ft. Included Angle.
3 7/16"
(a)
I. STANDARD SPECIMEN.
0.480"
d
LAYOUT OF SPECIMENS (b), (c), (d), (e), (f), (g).
See Enlarged Views for Notch Details.
0.038"
60°
0.01" R.
d = 0.404"
(b)
45°
0.038"
1/64" R.
d = 0.404"
(c)
0.023"
45°
Made with Slightly Blunted Tool
d = 0.434"
(d)
45°
0.076"
1/64" R.
d = 0.328"
(e)
1/64" R.
d = 0.449"
(f)
1/8" R.
d = 0.230"
(g)
0.094"
1/8"
1/8"
3/16"
3/4"
3/16"
3/16"
0.591"
0.094"
0.480"
(h)
3/16"
1 1/8"
3/16"
1/8"
1/8" R.
45°
0.687"
Taper 5/8" per ft. included Angle
0.716"
0.404"
6"
(i)
Drill No. I (0.272")
Depth 5/8"
Tap 5/16"-24 Threads per inch, U.S. Standard.
0.404"
45°
1/4" R.
3" R.
0.687"
0.966"
(j)
3/8"-24 Threads per inch, U.S. Standard.
(k)
1/4"-28 Threads per inch, U.S. Standard
(l)
0.375"
0.0271"
0.0052"
(m)
0.250"
0.0232"
0.00446"
(n)

FIG. 1.—Grooved and Threaded Fatigue Specimens.

These ends are cold rolled to $\frac{1}{2}$ in. in diameter. The chemical composition is given in Table I.

The corrosion specimens were taken from bars $\frac{1}{2}$ in. in diameter which were not analyzed at this time but are known to meet the specifications for S.A.E. 1045 steel.

METHOD OF TEST

Test Specimens:

The various types of grooves studied are shown in Fig. 1 which gives details of the repeated stress specimens. The types shown as *a, b, c, h, i,* and *j* were prepared in sets of 12 each in both S.A.E. 6130 steel and 25S alloy in order to give complete endurance curves. The types shown as *d, e, f,* and *g* were prepared in sets of four only, as it was believed that the endurance limit could be estimated very closely from four specimens after having obtained the complete curves on grooves of a generally similar type. This method proved quite successful and effected a great saving in cost of test specimens. Two tension test specimens were prepared of each groove. The tension test specimens are not shown, but it is sufficient to say they were threaded-end specimens of 2-in. gage length with the same dimensions at the notch as shown in Fig. 1.

In making heat-treated test specimens of various sizes it is always necessary to pay particular attention to the mass effect of heat treating. Therefore, the main types of grooves *a, b, h, i, j* were all made with the same root diameter in order to eliminate this factor. The purpose of these five specimens is to study the effect of the radius at the root of a groove of the form of a buttress thread. It was recognized, however, that test results would be affected by the ratio of depth of groove, d, to the diameter of specimen, D. To obtain a direct comparison of the effect of the radius, it would be necessary to hold the $\frac{d}{D}$ ratio constant. If the root diameter and $\frac{d}{D}$ ratios are to be held constant, then the outside diameter must be held constant. With these limitations, only the $\frac{1}{64}$ and $\frac{1}{16}$ in. radii at the bottom of the desired form of groove could be obtained, and it was very desirable to investigate the effect of larger radii. If the depth must vary it was decided to increase the depth the same amount as the increase in radius. By this method the distance from the center of the radius to the top of the groove is always the same (0.031 in.). In actual computation, the depth was obtained by adding the difference in radii between Fig. 1(*c*) and either of Figs. (*h*), (*i*), (*j*), to 0.048 in., which was originally to have been the depth of groove in Fig. 1(*c*). The drawing shows an actual depth of 0.038 in. The difference is neglible, as will become evident upon observing that the endurance limit of a groove of same radius but 0.076 in. deep (Table II, Column 5) is only 2000 lb. per sq. in. less.

The groove of Fig. 1(d) is in proportion to a $\frac{1}{4}$-in. pitch $3\frac{7}{8}$-in. diameter buttress thread used on propeller hubs. The groove e is an extra deep $\frac{1}{64}$-in. radius notch while f and g are ordinary radii.

Testing Machines:

The repeated stress tests were made in rotating beam fatigue machines of special type developed in this laboratory. The small specimens Figs. 1 (*a*), (*b*), (*c*), (*d*), (*e*), (*f*), (*g*), and (*h*) were run in a plain bearing machine shown in Fig. 2. In this type of machine, a short specimen is used (Fig. 1(*a*)), whereas in the Sondericker or Farmer type a very long specimen, which is difficult to work in the light alloys, is used. With the machine as constructed, considerable variation in length of specimen can be made without affecting the calibration of the machine or the set-up. The moment arm is permanently and accurately set when the machine is built and is never changed. This feature of course makes the apparatus much more accurate and reliable than the long specimen types. Operating conditions are ideal, the machine is quickly set up and convenient to handle. It runs extremely smoothly and quietly due to the use of plain bearings instead of ball bearings.

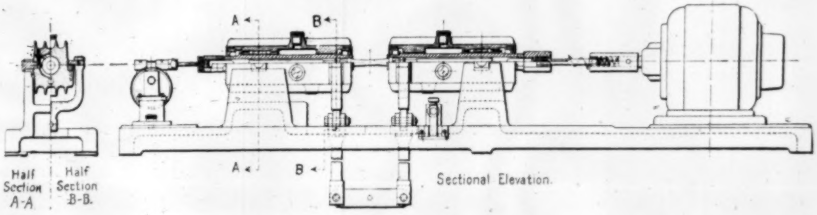


FIG. 2.—Short-Specimen Plain-Bearing Rotating-Beam Fatigue Machine.

The large specimens, Figs. 1 (*i*) and (*j*), were run in a machine of somewhat similar construction but much larger. It was built for testing tubes 1 in. in diameter and can be operated under very heavy loads.

Determination of Endurance Limit:

The values for endurance limits were determined by the generally accepted method of plotting stress-cycle curves. Where there were only four specimens available, a complete endurance curve could not be obtained. In such cases, specimens were not run longer than 20 million cycles, after which run the stress was raised. If the specimen then broke, this last test gave a point on the curve. However, in using these points to locate the curve, consideration was given to the probable increase in life over normal due to the effect of under-stressing (or coaxing). The lower part of Fig. 3 demonstrates this method. The dotted lines indicate the path of stress raising. The location of the point on the abscissa designates the actual number of reversals at the stress plotted—not the total number of cycles the specimen has passed through, including the low stress tests.

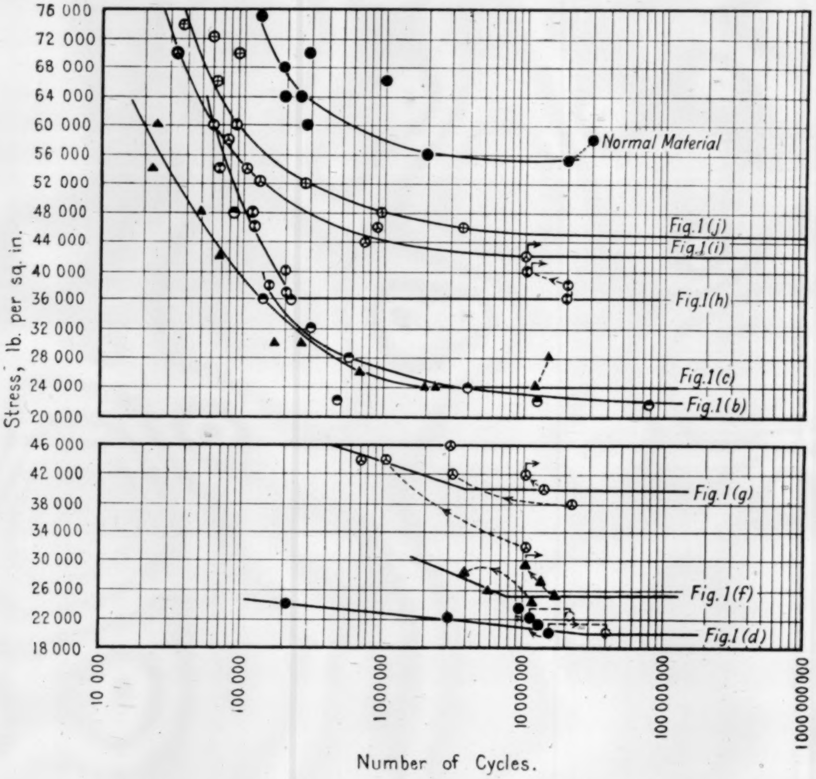


FIG. 3.—Results of Endurance Tests on 6130 Steel.

The endurance curves for the 25S alloy, in so far as they have been completed, are given in Fig. 4, and are somewhat more difficult to interpret. It appears that these curves show no definite "knee" or tendency to "flatten" below 100 million cycles. The slope of the curve for the standard specimens which represent the material free from any shape effect is quite sharp. The difference between the stress at 100 million cycles and at one billion cycles (if the curve were prolonged that far) is about 4000 lb. per sq. in. or a drop of $28\frac{1}{2}$ per cent. At a stress of 14,000 lb. per sq. in., one specimen failed at 60 million cycles. There are, however, two specimens still running at this stress, one has run 220 million cycles and the other 100 million cycles. The trend of the evidence seems to indicate that the curve will flatten at about 14,000 lb. per sq. in. This value has been temporarily selected as the endurance limit, but to be modified upon further evidence which may indicate a change to be necessary.

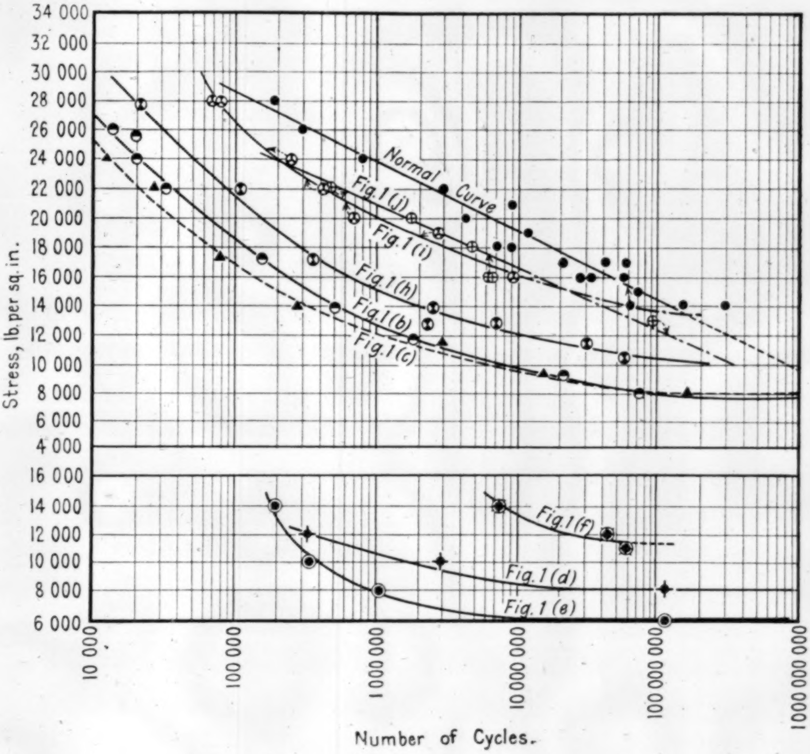


FIG. 4.—Results of Endurance Tests on 25S Alloy.

The endurance limits for the notched specimens of 25S have been chosen at the stress for 200 million cycles. In most cases, the curves have become so flat at this point that this method can introduce but little error.

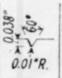
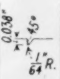
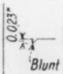
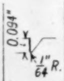
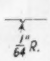
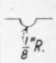
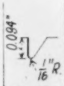
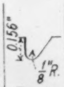
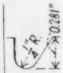
RESULTS OF TESTS

Effect of Shape, Depth, and Radius of Groove:

The detail results of the repeated stress tests are given in the endurance curves of Figs. 3 and 4. A summary and comparison of all test results is given in Table II.

The grooved specimens in every case develop a higher tensile strength then normally obtained for the metal on a standard test specimen. It appears that the intensified stresses set up due to the sudden change in section do not weaken the specimen, for it is actually stronger. This effect has been known for a long time. The failure of the intensified stresses to weaken the specimen in static tension is ascribed to the opportunity afforded for redistribution of the stress due to plastic flow at the high stress points long before the specimen has been subjected to the maximum load.

TABLE II.—RESULTS OF REPEATED-STRESS TESTS SHOWING EFFECT OF GROOVES.

	Standard Fig.1(a)	 Fig.1(b)	 Fig.1(c)	 Fig.1(d)	 Fig.1(e)	 Fig.1(f)	 Fig.1(g)	 Fig.1(h)	 Fig.1(i)	 Fig.1(j)
	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
EFFECT OF GROOVE ON 6130 STEEL										
Root Diameter, in.	....	0.404	0.404	0.434	0.328	0.449	0.230	0.404	0.406	0.404
Outside Diameter, in.	0.500	0.480	0.480	0.480	0.480	0.480	0.480	0.591	0 716	0.980
Proportional Limit, lb. per sq. in.	127 800	163 800	156 000	148 600	165 600	139 600	96 400	172 400	140 400	146 350
Tensile Strength, lb. per sq. in.	137 600	194 800	192 600	174 400	238 300	168 750	181 850	224 200	213 550	189 500
Elongation, per cent	18.5	2.5	2.0	6.0	1.0	....	1.7	3.0	3.7	3.5
Reduction of Area, per cent	65.0	9.9	11.1	18.9	9.0	23.7	37.6	43.5	20.6	30.4
Endurance Limit, lb. per sq. in.	55 000	21 000	24 000	20 000	22 000	25 000	40 000	36 000	42 000	45 000
Ratio of Proportional Limit Notch to Proportional Limit	....	1.28	1.22	1.16	1.29	1.09	0.754	1.355	1.009	1.145
Ratio of Tensile Strength Notch to Tensile Strength	....	1.416	1.400	1.267	1.731	1.226	1.322	1.531[1]	1.552	1.378
Ratio of Endurance Limit Notch to Endurance Limit	....	0.382	0.436	0.364	0.400	0.455	0.727	0.655	0.764	0.818
EFFECT OF GROOVE ON 25S ALLOY										
Root Diameter, in.	....	0.404	0.404	0.434	0.328	0.449	0.230	0.404	0.406	0.404
Outside Diameter, in.	0.500	0.480	0.480	0.480	0.480	0.480	0.480	0.591	0.716	0.980
Proportional Limit, lb. per sq. in.	19 360	28 325	31 200	28 730	41 420	23 790	35 800	35 260	38 030	32 590
Tensile Strength, lb. per sq. in.	59 875	62 600	64 475	54 750	74 150	63 925	70 400	77 975	75 325	70 025
Elongation, per cent	18.7	2.7	2.5	3.0	1.3	7.3	1.3	3.5	3.8	2.8
Reduction of Area, per cent	27.6	4.9	5.15	5.4	4.55	9.58	15.8	13.2	14.1	16.5
Endurance Limit, lb. per sq. in.	14 000	7 750	8 000	8 000	6 000	11 500	....	10 000	13 000	....
Ratio of Proportional Limit Notch to Proportional Limit	....	1.463	1.611	1.485	2.14	1.228	1.85	1.821	1.965	1.683
Ratio of Tensile Strength Notch to Tensile Strength	....	1.045	1.076	0.914	1.238	1.068	1.176	1.302	1.258	1.17
Ratio of Endurance Limit Notch to Endurance Limit	....	0.554	0.572	0.572	0.429	0.821	....	0.715	0.929	....

[1] Based on Tensile Strength = 146 500 instead of values of Column 1.

On the other hand, when subjected to repeated stresses the metal is decidedly weakened, for in every case the endurance limit was less than the normal endurance limit of the metal. In some cases, the endurance limit was less than 40 per cent of the normal value. With a $\frac{1}{4}$-in. radius the endurance limit was still only 80 per cent of the normal value. Under the action of repeated stresses there is little opportunity for the redistribution of stress. This may be partly due to the very small amount of strain incurred and the time element in applying the stress. The highly intensified stresses if located at weak grains or unfortunately oriented grains are still more effective in starting cracks.

Referring to Table II, it will be seen under columns 2 and 3 that there is practically no difference between the 60-deg. V groove and the 45-deg. groove with one straight side.

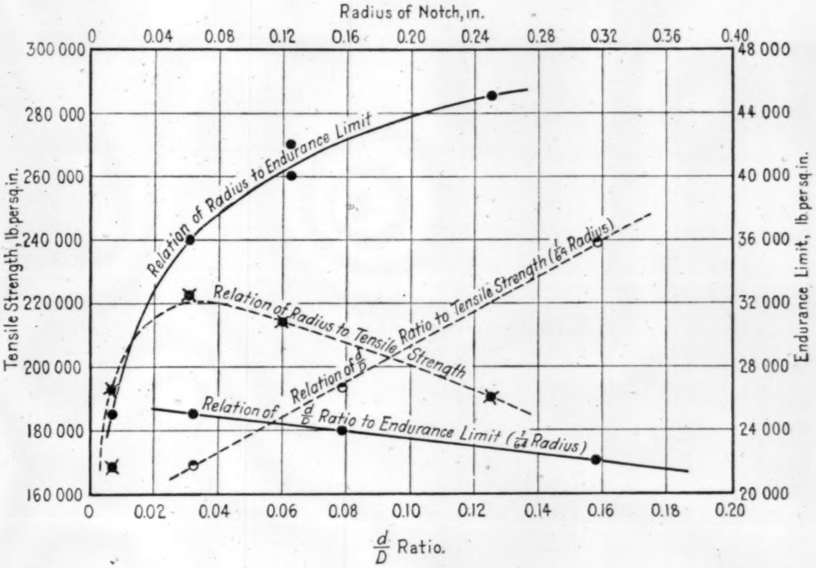


FIG. 5.—Effect of $\frac{d}{D}$ Ratio and Radius on Tensile Strength and Endurance Limit of 6130 Steel.

Considering only the tests on the 6130 steel, by doubling the depth of the $\frac{1}{64}$-in. radius groove of column 3 it is found (column 5) that the tensile strength increased about 24 per cent. The endurance limit, however, has fallen about 8 per cent. The effect of depth of groove is best shown in Fig. 5, which compares the three $\frac{1}{64}$-in. radius grooves of columns 3, 5, and 6. The effect of depth of groove may also be observed under columns 7 and 9 for the $\frac{1}{8}$-in. radius specimens. It appears that with the larger radius the weakening effect becomes less.

The effect of radius may also be seen in Fig. 5. In studying these results, however, it must be remembered that the $\frac{d}{D}$ ratio becomes greater and would tend to counteract the effect of increasing the radius.

However, from the observations of the two ⅛-in. radius specimens it is not believed that this will have any appreciable effect upon the ¼-in. radius specimen and the curve can probably be taken at face value.

The groove in column 4 is proportioned to a 3⅞-in. diameter, ¼-in. pitch buttress thread which is used on propeller hubs. It is evident that this shape considerably weakens the resistance of the metal to repeated stresses. There is one reservation to be made in applying this result. There is no available data to show that a reduction to 36 per cent of the normal endurance limit will persist in such a large diameter and groove as exists in the 3⅞-in. hub. It is doubtful that the large specimen will be as seriously affected, but to just how far these results can be proportioned is unknown until experimental data is obtained. Such an investigation would be well worth while.

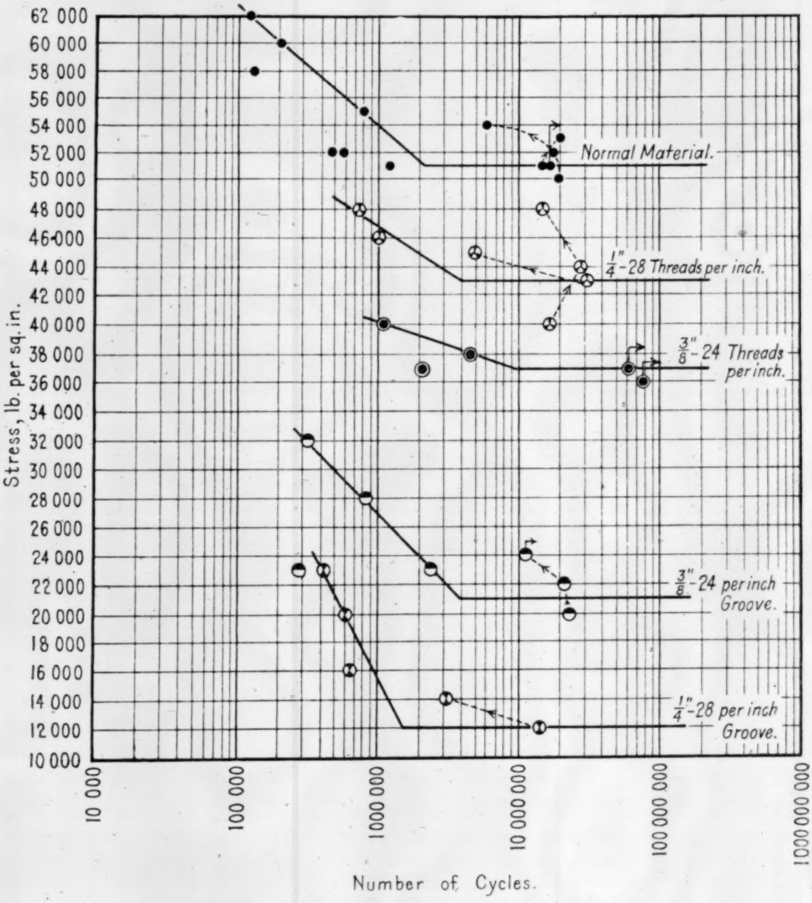


FIG. 6.—Results of Endurance Tests of Grooved and Threaded Specimens.

The results on the 25S alloy show that the grooves in general cause a somewhat greater increase in tensile strength and less weakening of the endurance limit than for the 6130 steel. It will be noticed that the normal endurance limit of this alloy is very low, only about $23\frac{1}{2}$ per cent of the tensile strength.

Effect of Threads and Radial Grooves:

From the peculiarities of certain service failures, it has been suspected there was some difference between the effect of the groove formed by a continuous thread and that of a radial groove of the same proportions. Tension and repeated stress specimens were prepared with $\frac{1}{4}$-in. diameter, 28 threads per inch and with $\frac{3}{8}$-in. diameter, 24 threads per inch U. S. Standard form thread. Specimens with a radial notch of both forms and proportions were also prepared. The endurance specimens are shown in Figs. 1 (*k*) and (*l*).

TABLE III.—RESULTS OF TESTS ON THREADED AND GROOVED SPECIMENS.

	Plain	$\frac{1}{4}$-in. 28 Threads per Inch		$\frac{3}{8}$-in. 24 Threads per Inch	
		Notch	Thread	Notch	Thread
Root Diameter, in.	....	0.203	0.203	0.321	0.321
Outside Diameter, in.	....	0.250	0.250	0.375	0.375
Proportional Limit, lb. per sq. in.	58 850	84 850	61 760	83 450	74 200
Tensile Strength, lb. per sq. in.	140 600	166 000	140 600	165 000	156 300
Elongation, per cent	4.0	0	2.5	0	3.25
Reduction of Area, per cent	14.9	....	....	....	....
Endurance Limit, lb. per sq. in.	51 000	12 000	43 000	21 000	37 000
Ratio of Tensile Strength Notch to Tensile Strength	....	1.181	1.000	1.172	1.112
Ratio of Endurance Limit Notch to Endurance Limit	....	0.235	0.844	0.412	0.726

The results of the endurance tests are given in the curves of Fig. 6. A comparison and summary of tests results is given in Table III.

While both the grooved and threaded specimens generally show a higher tensile strength than the normal strength of the steel as obtained on a standard test bar, the excess strength is not very great. In fact the $\frac{1}{4}$-in. 28 threaded specimen is about the same.

The endurance test results, however, show quite a different effect. The notched specimens show considerable drop in the endurance limit, the $\frac{1}{4}$-in. 28 notched specimen falling to 23.5 per cent of the normal value for the steel. The $\frac{3}{8}$-in. 24 thread notch is much stronger than the $\frac{1}{4}$-in. 28 thread. This may be due to a lower $\frac{d}{D}$ ratio. A marked difference, however, is found between the radial notched and continuously threaded specimens, for the threaded specimens are very much stronger, rising to about 72 to 85 per cent of normal strength.

These results should be carefully considered as they show that the study of the effect of threads must be made with continuous threads and not with radial notches of equivalent form. In this connection any further study of the effect of size of specimen as suggested under the section on effect of shape of grooves should give consideration to these test results.

The increased strength of the continuously threaded section under repeated flexural stress may be due partly at least to the helical path of the groove as it travels along the rod. This causes the minimum section (groove) to lay at an angle of less than 90 deg. to the axis of the beam. Therefore a section cut perpendicular to the axis of the beam is greater than the minimum section measured at the root of the thread.

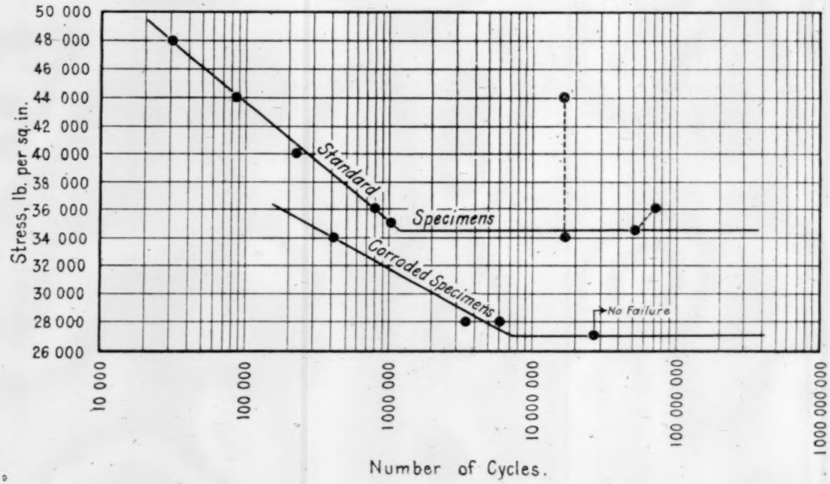


FIG. 7.—Effect of Corrosion on Endurance Limit.

Effect of Corrosion:

It is a general observation that corrosion does not seriously affect the tensile strength of steel unless the corrosion be exceptionally heavy and the steel of very thin gage. Corrosion acts to produce pits in steel, and from the results of the tests showing the effects of notches it was suspected that under repeated flexural stresses, these pits (which are fine notches) may be sufficient to cause a measurable reduction in the endurance limit. Test results confirm this suggestion.

Samples of 1045 steel were subjected to a salt spray (20-per-cent sodium chloride solution in water) for 40 days. They were then removed and carefully cleaned of the rust in so far as it was practical without damaging the surface of the specimen. Specimens were examined under the microscope for pitting and it was found that the average depth of pit was in the order of 0.0015 in.

The results of the endurance tests are shown in Fig. 7. Test results are summarized in Table IV.

The test results show that the degree of corrosion obtained by the 40-day test was sufficient to cause a marked reduction (21.7 per cent) in the endurance limit. It is of course difficult to measure or compare degrees of corrosion, but it is seen that the pitting incurred here is hardly excessive. The effect of the corrosion on the endurance limit, however, was apparent but it cannot be definitely stated as to whether the weakening is due to the actual presence of the pits (as a basis for developing intensified stresses and starting fine cracks) or possible corrosion of susceptible grains or grain boundaries or some other cause not readily apparent.

TABLE IV.—RESULTS OF TESTS SHOWING EFFECT OF CORROSION.

Tensile Strength, lb. per sq. in	76 000
Endurance Limit, lb. per sq. in	34 500
Ratio of Endurance Limit to Tensile Strength	0.454
Tensile Strength after Corrosion, lb. per sq. in	75 800
Endurance Limit after Corrosion, lb. per sq. in	27 000
Reduction in Endurance Limit Due to Corrosion, per cent	21.7

What the effect of various types of corrosion, such as prolonged exposure under various conditions, excessive pitting, acids, etc., may have upon the endurance of metals is unknown and there is very little information available in the literature. The tests described are very general and do no more than point out certain possiblities and demonstrate the inadequacy of the tension test.

SIGNIFICANCE OF THE REPEATED-STRESS TEST

It has just been shown that the results of the repeated-stress test bear no definite relation to the results of the tension test in the presence of such factors as grooves, threads, and corrosion. In all cases, the results of repeated stress tests showed that these factors were far more detrimental to the life of the material than would be inferred from the results of the static tension test.

As the underlying cause of weakening of notched or grooved specimens is generally assumed to be the presence of intensified local stresses, it might be inferred that a determination of the magnitude of these stresses would offer a solution as to the effect of the notch. The determination of the magnitude of these stresses by mathematical analysis is difficult even for simple shapes. The photo-elastic experimental method of Coker or Soap Film method of Griffith is much more desirable for the difficult shapes, and both these methods have been used very successfully for determining the intensity of local stresses.

It has, however, been shown by the author[1] that even with the value of the intensified stresses known we are hardly any further advanced than before so far as determining the actual quantitative effect upon the service life of the material or part. This is due to the fact that metals respond differently to the imposed stresses and no mathematical or experimental analysis as yet has been devised to accommodate the results of the concentrated stress analysis to the particular material. A mathematical analysis of the concentration of stress in the 60-deg. V notch in Fig. 1(*b*) has been made.[1] If it is to be assumed that this intensified stress cannot be exceeded without incurring failure, then the endurance limit should be reduced about 78 per cent (to 22 per cent of normal value). It is evident that this reduction does not obtain on either the 6130 steel or the 25S alloy.

The author has paid particular attention to the effect of elongation and has shown[2] that this property has no effect at all upon the results of notched specimens.

Accordingly then it is quite apparent that neither the tension test, or mathematical or experimental anaylses of stress concentrations, in their present stage of development has much to offer towards the solution of the effect of various concentrated stress-producing factors upon the resistance of metals to repeated stresses. The repeated-stress test, or fatigue test, or endurance test, whatsoever it should be called is the nearest approach to service conditions and is of far greater value than any test yet devised for determining the general usefulness of materials. To be included in this generalization are repeated stress tests at both high and low temperatures.

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[1] R. R. Moore, "Resistance of Metals to Repeated Static and Impact Stresses," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 566 (1924).

[2] R. R. Moore, "Some Fatigue Tests on Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 66 (1925).

DISCUSSION ON FATIGUE OF METALS

Mr. McAdam.

Mr. D. J. McAdam, Jr. (*supplementary comments made in oral presentation of his paper*).—In the effort to limit the size of the paper some desirable qualifying comments were omitted from my discussion of the effect of ductility on corrosion-fatigue. The effect of ductility on corrosion-fatigue limits is probably greater than its effect on the endurance limit. The effect of ductility, however, is overshadowed by the effect of varying corrosion resistance.

Though microscopic cracks, such as those shown in Fig. 12, are numerous in most carbon and alloy steels after failure under corrosion-fatigue, they were not found in the stainless iron unless the corrosion-fatigue limit was considerably exceeded; even then, they were few.

Whether or not corrosion-fatigue cracks originate in the nonmetallic inclusion or pores in the metal, their chief progress is probably along intercrystallin boundaries. Preliminary investigation of ingot iron indicates that for this material corrosion-fatigue failure is at least partly intercrystallin.

It seems probable that the corrosion-fatigue limit of metals depends chiefly on the corrosion-resistance and strength of the predominant phase, especially at the intercrystallin boundaries. Apparently the corrosion-fatigue limit depends only slightly on the fineness and distribution of a second phase, such as the carbides in steel.

Mr. H. F. Moore.

Mr. H. F. Moore[1] (*presented in written form*).—Mr. McAdam's paper gives some very valuable comments on the general characteristics of graphs of fatigue tests and rather startling test results on the effect of corrosion on fatigue strength.

As noted by him in the third and fourth paragraphs of page 237 and as shown in his Fig. 7, there may be a considerable change in the upper part of a fatigue test graph without much change in the endurance limit. This fits in with tests on monel metal made at the University of Illinois. Tests of two lots of monel metal as received—hot rolled—gave graphs with no appreciable curvature although tests were run to 800 million cycles of stress. Tests of annealed specimens from one lot showed a fairly well-marked endurance limit, with a distinct change of slope in the diagram.

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. H. F. Moore. The history of this monel metal leads to the suspicion that these particular lots either had high internal stresses left by rolling or perhaps there were actual sub-microscopic cracks in the metal. This leads to the suggestion that in addition to the various factors which Mr. McAdam has listed as affecting the shape of the fatigue graph there may be added the state of internal stress and the mechanical soundness of structure of the metal.

The writer would like to call attention to the fact that while different machines and different specimens give different shaped graphs for fatigue tests, careful tests in different laboratories show a very general agreement as to the values of endurance limit obtained.

Mr. McAdam's discussion on extrapolation of fatigue test graphs is specially worthy of study. While it is not yet certain whether the whole graph for a fatigue test is a continuous function or whether there is a discontinuity at the endurance limit, Mr. McAdam has shown a very satisfactory method of making a close estimate of endurance limit from a graph which has become nearly but not quite horizontal. In fact, a simple graphical extrapolation usually gives satisfactory results.

About all that can be said of Mr. McAdam's results on corrosion-fatigue are that they are rather startling. Some check tests made on larger specimens would be of interest to show whether the minute cracks that are formed damaged large pieces as severely as small pieces. Mr. McAdam's tests and the tests reported by Mr. R. R. Moore demand that attention shall be paid to the effect of corrosion on fatigue strength.

A striking feature of the paper by Mr. R. R. Moore is his demonstration of the fact, already known but not very generally recognized, that sudden changes of form—"stress-raisers" as Mr. Gillett calls them—do not reduce the fatigue strength of a machine part nearly so much as is indicated by the mathematical theory of elasticity. Moreover, different metals are affected in different degrees by such "stress-raisers." Tests at the University of Illinois seem to indicate that alloy steels are affected rather more by local "stress-raisers" than most other steels—a result in line with Mr. McAdam's corrosion-fatigue results. Mr. R. R. Moore's results on the relative effect of a screw thread and a groove became known to the writer some weeks ago and check tests on soft steel rods were run at the University of Illinois. While the tests are not quite finished, the results check Mr. R. R. Moore's results in a general way and in addition indicate that a series of grooves equal in length and pitch to the screw thread and cut with the same tool are intermediate in weakening effect between the screw thread and the single groove.

Mr. H. F. Moore. Mr. Irwin's paper supplements effectively his work reported last year and in the writer's opinion establishes the reliability of the simple and inexpensive rotating-beam machine for fatigue tests.

The writer believes the Society is to be congratulated on these three papers, each making one or more distinct contributions to our knowledge of the fatigue of metals.

Mr. Gillett. MR. H. W. GILLETT[1] (*presented in written form*).—It would be very interesting if Mr. McAdam would give micrographs showing whether the cracks due to corrosion combined with repeated stress are propagated through or between the crystals. Repetition of some of the experiments, using a potassium-dichromate solution instead of water, to avoid rusting while still accomplishing cooling, would also be expected to give interesting results.

The observation that elongation at maximum load is the best index of the ratio of the endurance limit under corrosion to that with no corrosion, is doubtless to be taken as referring only to this specific set of observations. If the cracks develop in an intercrystallin fashion one would expect that resistance to intercrystallin attack as well as to the spreading of cracks after they start, would be involved. In light aluminum alloys, susceptibility to intercrystallin attack varies with composition even though the alloys have similar ductilities. "Corrosion-fatigue" must involve both chemical and physical properties and without evidence based on other classes of material than steels alone it would be premature solely to connect performance with a purely physical property. It is believed, therefore, that Mr. McAdam's comment ought, in the absence of further proof, to be restricted for the present to the eight steels tested, although in his final paragraph it would appear that he expects it to apply to non-ferrous alloys also.

Mr. Lessells. MR. J. M. LESSELLS.[2]—The results given by Mr. R. R. Moore on effect of grooves call for some comment. It is now generally known that any stress concentration will not affect the tensile strength of a ductile material since the material by virtue of its ductility is able to adapt itself by local yielding. If the material be brittle it is expected that due to the lack of ability to yield, any stress concentration will lower the tensile strength. In fatigue, however, the case is different and here we would expect stress concentration to lower the endurance limit for both ductile and brittle material, the lowering being greater for the latter material.

If we refer to the tensile results given for steel 6130 Figs. 1(*h*), 1(*i*) and 1(*j*) in Table II of the paper, it will be seen that as the

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[1] Chief, Division of Metallurgy, U. S. Bureau of Standards, Washington, D. C.

[2] Engineer in Charge, Mechanics Section, Research Laboratory, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Mr. Lessells. radius of the groove increases the tensile strength decreases. This seems difficult to explain and calls for some comment by the author. The results obtained for the endurance limit show an increase with increase in groove radius which is what must be expected. This material, it should be noted, is, from the tensile results on the normal test bar, a very ductile material. The same criticism can be made of the results obtained on alloy 25S for the same models, namely, an increase in groove radius lowers the tensile strength, which is again difficult to explain, and increases the endurance limit, which is to be expected.

It may be of interest in this connection to observe that the writer with S. Timoshenko has recently made some tension tests on notched test bars cut from the same bar of low-carbon steel and found that as the notch, in this case rectangular, is reduced in width from 1 to $\frac{1}{32}$ in., the tensile strength increases and even in the case of the narrow notch the fracture occurred, not at the neck where stress concentration was present but in the middle of the notch. This is, from the above reasoning, what can be expected. The increase in tensile strength arises of course from the suppression of lateral contraction due to the decrease in groove width.

It is because this phenomenon of stress concentration is receiving more and more attention by designers that this criticism is offered. It seems some explanation of unexpected results must be forthcoming.

Mr. R. R. Moore. MR. R. R. MOORE.—Mr. Lessells' discussion has helped to emphasize some very interesting and valuable factors of stress concentration.

If interpreted correctly his own test results as described for the rectangular notch agree with the author's instead of disagreeing as Mr. Lessells has interpreted them. It is true that the author's tests show a decrease in tensile strength with increase in radius of the notch. This is due to the greater opportunity for lateral contraction of the material which is necessary to develop the normal (or "engineering") value for tensile strength. Mr. Lessells quotes results which show an increase in tensile strength with decrease in notch width which is the same as saying a decrease in strength with increase in notch width. Evidently the increase in notch width acts the same as the increase in radius in the writer's tests, and the two sets of tests are in agreement.

It is interesting to note that the "lateral contraction" factor is of greater importance than the sharp corner of the square shoulders, as Mr. Lessells points out in noting that the fractures occurred in the middle of the notch rather than at the sharp corner.

Mr. H. F. Moore.

MR. H. F. MOORE.—In connection with the theoretical and the effective raising of localized stress by holes, grooves, screw threads, etc., I should like to refer briefly to a method of studying the effect of such "stress-raisers" which has been developed in a small way at the University of Illinois by the speaker and Mr. R. E. Peterson. This method consists in determining the relative ultimate strength of two series of specimens, one series containing the stress-raiser to be studied, and the other being as free as possible from stress-raisers of any kind. Both series of specimens are made of some brittle material, and the material so far found most suitable is pottery plaster, which can be easily molded, can be machined after molding, can be produced of uniform quality, and has a stress-strain diagram which deviates but little from a straight line right up to rupture, a fact which was pointed out some years ago by W. A. Slater.

The tests made so far check the general findings of Mr. R. R. Moore in his paper, and of H. F. Moore and Jasper in *Bulletin No. 152* of the Illinois Engineering Experiment Station, namely, that the effect of stress concentrations on strength of a material is less than the effect predicted on the basis of theoretical stress analysis. So far the plaster model tests give stress-concentration factors not differing very widely from those given by repeated-stress tests of steel specimens.

Mr. Mochel.

MR. N. L. MOCHEL.[1]—Figure 12 of Mr. McAdam's paper and several of the slides he used show the fatigue cracks starting from small pits. It is stated that the monel metal used "as shown by metallographic examination, was of excellent quality and with few non-metallic inclusions." This monel metal showed no effect of corrosion-fatigue. Now it is well known that this alloy and similar alloys may be rich in inclusions such as magnesium-sulfide which are readily washed out by the action of water alone. What would be the effect of corrosion and fatigue on such monel metal, in which many pits existed where the inclusions had been removed?

Then again, in the matter of the steels and especially the stainless iron, non-metallic inclusions would be the nuclei for the starting of corrosion, and pits would develop. A "clean" steel or stainless iron would have fewer pits from which fatigue cracks could start, whereas a "dirty" steel would have many such pits. Might we expect poorer results from a "dirty" steel?

Mr. McAdam.

MR. MCADAM.—In Mr. Irwin's excellent paper, the tensile strength of his "annealed" nickel is given as over 80,000 lb. per sq. in. The nickel that we have tested in the fully annealed con-

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[1] Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., Lester Station, Philadelphia, Pa..

Mr. McAdam. dition has a tensile strength of 60,000 to about 75,000 lb. per sq. in. and the endurance limit is lower than for the nickel used by Mr. Irwin. It seems possible, therefore, that the nickel used by Mr. Irwin was not in the fully annealed condition.

I was much pleased with Mr. H. F. Moore's remarks. In general the results obtained in the two laboratories have been in good agreement.

In reply to Mr. Mochel's questions, I should say that variation in the force of the water stream seems to have little effect on the results. A small slow stream of water causes more rapid rusting and the damaging effect is therefore slightly greater than the effect of a larger stream that fully surrounds the specimen. As shown in Fig. 1, a monel metal specimen tested for nearly 2,000,000 cycles in water at about 65° F. broke at about the same position as did specimens tested in air.

The effect of quantity of non-metallic inclusions has not been investigated. I do not expect, however, to find great variation in the corrosion-fatigue limit with variation in quantity of inclusions. In most steel specimens that have failed by corrosion-fatigue failure, the corrosion spots surrounding inclusions are numerous. In the stainless-iron specimens, however, only a few spots were found in highly stressed specimens, and none in specimens stressed slightly above the corrosion-fatigue limit.

Mr. Thum. MR. E. E. THUM.[1]—I believe that most engineers will be interested in Mr. Irwin's conclusions. They also will undoubtedly be disquieted by Mr. McAdam's discovery that a very minor amount of corrosion operating for a very few hours will cause such a large difference in the expected results of rotating-beam fatigue tests. Mr. McAdam's paper emphasizes again the pronounced effect the condition of the surface has upon the rotating-beam endurance limits. The methods of testing used by Mr. Irwin will doubtless be influenced to a minor extent by surface corrosion, since a localized yielding there will often be relieved by a redistribution of stress over the entire cross-section, and not remain the region of highest stress concentration.

If that be true, it seems to me that the direct stress method of Mr. Irwin (and it is also used abroad to a considerable extent) is a better indication of the true ability of the metal to resist alternating stress than the rotating-beam method. Of course we cannot forget the fact that many pieces liable to fail in fatigue operate as a rotating beam, and also that the operation of some pieces will require a very high degree of perfection on the surface; nevertheless, many engineer-

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[1] Manager, Technical Publicity Department, Linde Air Products Co., New York City.

Mr. Thum.

ing structures are arranged so that the members are expected to act in direct compression and tension. Consequently Mr. Irwin's results are directly usable in such computations.

A fact not to be neglected by one theorizing on the nature of fatigue is also evident: that fatigue limits in tension and bending are the same only so long as the surface of the rotating beam represents the average condition of the cross-section of the piece.

It seems to me that fatigue experiments are indicative of almost undefinable "quality" in the metal. Apparently minute defects in the metal have a great influence on the result. Is there not also food for thought in the observation that there is also a general relationship between the quality of the metal and the number of minute internal voids? Makers of the highest class of steel for ordnance have a term which they use to represent the best steel that they can make. They call it "tight steel." Such steel is not easy to forge; it is rather delicate, but nevertheless a maker of fine steel will tell you that a tight steel is a superfine steel. As the quality of steel drops, they say the steel becomes somewhat "loose." One cannot pin a furnaceman down as to just what he means by "tight" steel and "loose" steel, but I wonder is it not, in general, an indication that the so-called tight steel, the very high-quality steel, the steel which stands up excellently in fatigue, either in rotating beams or in direct stress, is a steel which actually is tighter and contains fewer microscopic or submicroscopic holes—not inclusions, but actual voids?

Mr. McAdam.

Mr. McAdam.—Mr. Thum, in his comparison of the rotating beam or cantilever method with the direct tension-compression method used by Mr. Irwin, appears to lose sight of the fact that results of endurance tests by both methods are in good agreement. Investigation of fatigue of metals by rotating beam or cantilever method, therefore, gives results of direct practical application. A comparison of corrosion-fatigue results obtained by both methods is yet to be made. Very few machinery parts, however, are subjected to a repeated stress range of pure tension-compression. Bending or torsional stresses are usually present. Moreover, the great majority of machinery parts and a large proportion of structural parts, such as plate and sheet material, are subjected to repeated bending. Investigation of corrosion-fatigue by the rotating-bend method, therefore, gives results of direct practical application.

Mr. Jeffries.

Mr. Zay Jeffries[1] (*by letter*).—I wish to extend a word of commendation to Mr. McAdam both because a subject of great value has been brought to the attention of those interested in engineering

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[1] Consulting Metallurgist, Aluminum Co. of America, Cleveland, Ohio.

Mr. Jeffries. materials and because of the large amount of information which has been presented in a relatively small amount of space.

A point of particular interest in Mr. McAdam's results is the extension of the apparent load under repeated stresses to a value above the static tensile strength of the material. Mr. McAdam assumes that the stress-strain proportionality is not maintained at the higher stresses and consequently that the actual stresses are not as great as the calculated stresses, and probably not greater than the static tensile strength. It should be kept in mind that the values for both tensile strength and elongation are not fundamental constants of the material, but depend on the breaking of the specimens in a certain manner. In materials with high reduction of area the measured tensile strength is determined to a considerable extent by the reduction of area and the same is true of elongation. For example, a cold-drawn wire which shows an elongation in the tensile test of only 1 per cent in 2 in. may be drawn through a die to several times its original length without rupture. The elongation therefore is determined by the manner of applying the stress.

The same is true, but to a lesser extent, of the tensile strength. This has been brought out many times in the literature. Examples were given in the paper by R. R. Moore showing a markedly greater tensile strength per unit area in notched or threaded specimens than with specimens of the same material with smooth surfaces. In the latter case there can be no question as to the actual higher unit stress in the notched material than obtained in the unnotched material. The measured tensile strength in the unnotched specimens is the value obtained when the specimen is allowed to neck down at the point of fracture. This necking favors the gradual rupture of the metal. In the specimens used by Mr. McAdam it might be easily possible to stress a small area of the metal in tension higher than the static tensile strength of the material owing to the fact that the nature of the loading is such as to prohibit fracture by the necking down of a restricted area of the specimen.

Mr. Speller. MR. F. N. SPELLER[1] (*by letter*).—The influence of corrosion on the endurance limit of metals as propounded by Mr. McAdam is very suggestive. The amount of corrosion as measured by the penetration measurable from the surface of the test piece is apparently altogether too small to account for the marked reduction in the endurance limit. It is well known that when iron is subject to chemical action in various solutions, more or less hydrogen is formed and that this element is either evolved as gas dissolved in the solution or taken up by the metal.

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[1] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

Hydrogen penetrates iron with considerable rapidity, and as Williams and Homerberg have pointed out, may react with the non-metallic inclusions along the grain boundaries so as seriously to affect the bond between the grains. The fact that Mr. McAdam found in his tests that samples which were corroded before being subject to stress do not show such a low endurance limit as the samples that were corroded while under test, would point to such an explanation, as the hydrogen does not stay in the metal long after the metal is removed from the solution. In other words, it would seem that the hydrogen may penetrate and react with the metal along the grain boundary far ahead of the corrosion observed from the surface of the metal. **Mr. Speller.**

MR. J. B. KOMMERS[1] (*by letter*).—Mr. McAdam has added another brilliant discussion to his list of papers on fatigue of metals, and is to be congratulated on his latest contribution. **Mr. Kommers.**

While the discussion of the effects of various factors on the shape of the *S*—*N* curve at high stresses is of interest, it should be recalled that any conclusions drawn with respect to high stresses may not hold at stresses near the endurance limit.

Mr. McAdam's studies on the effect of heat interchange between the specimen and its environment, and on the effect of corrosion-fatigue, are of great importance in helping to make clear how fatigue failure may be influenced by certain perhaps unsuspected factors. These studies also show the care which any investigator in fatigue of metals must use in order that he may not draw the wrong conclusions from his results. It is evident that the experimenter must be very careful in order that the variables in the experiment may be reduced as nearly as possible to a single one. All students of the subject will be grateful to Mr. McAdam for demonstrating so clearly the effect of certain factors which hitherto have not been so fully examined.

MR. MCADAM (*author's closure by letter*).—In addition to the two possible causes that Mr. H. F. Moore gives to account for the unusual straightness of one of his monel metal graphs, I would suggest as a possible cause, corrosion-fatigue. Recent experiments have shown that monel metal is unexpectedly sensitive to corrosion-fatigue and that the influence of corrosion-fatigue is not usually apparent until a specimen has endured millions of cycles. **Mr. McAdam.**

Mr. Moore expressed some doubt whether the graph for a fatigue test is a "continuous function." In regard to this, I would say that investigation of a great variety of ferrous and non-ferrous metals at the Naval Experiment Station has shown no evidence of a discon-

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[1] Associate Professor of Mechanics, University of Wisconsin, Engineering College, Madison, Wis.

Mr. McAdam.

tinuity in the stress-cycle graph. The graphs in the present paper seem to strengthen the evidence in favor of continuity. The fact that many of Mr. Moore's graphs may be represented fairly well by two intersecting straight lines has suggested to him the idea of discontinuity. In the present paper, however, it has been shown that the form of the graph depends on the form of the specimen. As shown in Fig. 1 of my paper, the graphs obtained at the Naval Experiment Station consist of a curve running upward into a reverse curve. In most of the graphs in Fig. 1, this reversal of curvature is conspicuous. In a few graphs, however, the curvature in the two portions of the graph at their junction is so slight that these two portions unite to form a nearly straight sloping line. This is especially noticeable in the graph for Material DYA-8. Such a graph, however, must be regarded as due to the accidental balancing of two opposing curvature tendencies. Apparently the form of Mr. Moore's specimens was such as to favor this fortuitous shape of graph.

I have emphasized the probable continuity of the stress-cycle graph chiefly on account of its practical importance. If the stress-cycle graph is not a continuous function it cannot be extrapolated. The evidence presented in the paper, however, seems to indicate that the graph can be extrapolated.

Mr. Gillett is correct in his idea that the physical properties of a metal constitute only one factor in corrosion-fatigue, the other factor being corrosion resistance. In the steels investigated the latter factor does not vary greatly; so attempt was made to find a factor based on physical properties. Of the physical properties measured, the only one that appeared important as a factor in resistance to corrosion-fatigue was ductility. In mentioning the possible influence of the high ductility of some non-ferrous metals, it was not intended to imply that the other factor, corrosion-fatigue, is not of importance. A qualifying statement to this effect was given in my oral presentation of the paper. With further recent progress in the investigation, however, ductility as a factor in resistance to corrosion-fatigue appears less important and corrosion resistance appears even more important. Other physical properties possibly involved in resistance to corrosion-fatigue are discussed in a paper[1] giving results of continued investigation of this subject.

Microscopic examination of failed corrosion-fatigue specimens of a variety of steels has not revealed any purely intercrystallin fracture.

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[1] D. J. McAdam, Jr., "Corrosion-Fatigue of Metals as Affected by Chemical Composition, Heat-Treatment and Cold-Working," paper presented at the Annual Convention of the American Society for Steel Treating, Chicago, September 21, 1926.

Mr. McAdam.

In its path from one inclusion to another, however, a crack sometimes deviates and for a short distance follows intercrystallin boundaries. It is possible that at lower cycle frequency the higher ratio of corrosion to fatigue may increase the tendency toward intercrystallin fracture.

Jeffries' view that the actual stress in some of the graphs in Fig. 1 is possibly above the tensile strength of the material is in agreement with mine. In arguing that the actual stress in the upper part of these graphs is below the nominal stress, I had not intended to imply that the actual stress may not exceed the tensile strength. For example, in a specimen tested at a nominal stress of 175,000 the actual stress is undoubtedly below 175,000 but may be above 135,000 lb. per sq. in., the tensile strength of the material.

Mr. Speller's theory that the weakening effect of slight aqueous corrosion is due to absorption of nascent hydrogen is of considerable interest. This theory would probably not be inconsistent with the fact that the corrosion-fatigue limit varies greatly with intensity or corrosion or with corrosion resistance. For example, in salt water the corrosion-fatigue limit of steel is much lower than in fresh water. The fact that non-ferrous metals are subject to corrosion-fatigue, however, is not readily explained by the hydrogen theory.

In the September issue of *The Metallurgist* (supplement to *The Engineer*) is a review of my paper, which emphasizes the fact that I have omitted reference to a paper by Haigh.[1] In some experiments described in Haigh's paper, brasses and bronzes were subjected to alternating stress while in contact with corrosive agents such as ammonia, salt water and hydrochloric acid. He found slight lowering of the stress-cycle graph when Muntz metal and Naval Brass were etched with ammonia, but found no such lowering with the other combinations of metal and corrosive agent. Haigh's experiments were extended to only about 1,500,000 cycles, so the corrosion-fatigue limit for brass and ammonia was not obtained.

Haigh ascribed the effect on brass to the well-known strong attack of ammonia on the Beta constituent of brass. He points out that the damaging effect of ammonia is not produced unless corrosion and fatigue are simultaneous.

Although Haigh suggests the possibility that corrosion may affect unfavorably the endurance properties of metals in service, his results gave no indication of the great and general effects of even slight corrosion when simultaneous with fatigue. Haigh's experiments, how-

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[1] B P. Haigh, "Experiments on the Fatigue of Brasses," *Journal*, Inst. of Metals, 1917, No. 2, pp. 55–86.

Mr. McAdam. ever, made less impression than they deserved. Seven years later Gough's comprehensive book, "The Fatigue of Metals," made no mention of corrosion. Haigh's experiments had escaped my attention, and the absence of any reference to corrosion in Gough's 1924 book led me to believe that no important investigation of corrosion-fatigue had been made.

RECENT DEVELOPMENTS IN THE USE AND FABRICATION OF CORROSION-RESISTANT ALLOYS

By T. Holland Nelson[1]

Synopsis

In this paper are reviewed the various types of chromium-iron corrosion-resistant alloys available, particular mention being made of the possibilities of their application for various purposes. Special mention is made of the recent application of the low-carbon intermediate-chromium high-silicon material in the construction of large tanks for use in connection with nitric acid.

It is pointed out that in many cases where a variety of so-called stainless irons or steels meet laboratory tests, so far as corrosion resistance is concerned, many of these are limited for use due to the fact that various practical fabricating problems render their use, if not impossible, more difficult than similar materials possessing equal corrosion resistance, but which due to metallurgical differences, so far as chemical composition is concerned, make them more capable of being produced by generally accepted practice in the various forms called for in structural work of this nature.

The author states that there is no one chromium iron to meet all requirements and it is his hope that the paper will prove helpful to possible users of chromium iron in the sense that such users would very carefully study their problems and the various types of material available before launching into the fabrication of units calling for many and complex shapes.

It is now some fourteen years since stainless steel actually made its appearance. It is not the author's intention in this paper to cover matters relating to corrosion-resistant alloys which have been repeatedly brought out by various authors during this period. However, practically all the technical papers on this subject have dealt with it strictly as an academic problem. For a long time there has been no serious development of any magnitude, so far as fabrication is concerned, which has led to the gathering together of data which the author feels has been sorely needed by the prospective user of these various metals. It is, of course, understood that the cutlery industry is excluded from these remarks.

In discussing stainless and corrosion-resistant materials, the author has always found it easier to place them in three classes:

1. The original stainless steel of Brearley, with 9 to 16 per cent of chromium;

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[1] Consulting Metallurgist, Ludlum Steel Co., Watervliet, N. Y.

2. The later developments with over 16 per cent of chromium, with or without other alloy additions;

3. The nickel-chromium alloys which vary considerably in proportions of nickel and chromium.

As the author in this paper intends to deal more particularly with the fabrication of materials for corrosion resistance, it is not intended to refer more than casually to the heat resistance of these various metals although they are finding at the present time almost as heavy demand in the field of heat resistance as they are in the field of resistance to corrosion.[1 2 3]

1. *Stainless Steel:*

In reviewing the above three classes it should be understood that their applications are in many cases easily classified by their physical properties, or the ease or difficulty encountered in the fabrication of the metal. For instance, a typical analysis of stainless steel made under the original patent of Brearley, and available to-day on the open market, would come, say, under the following range of analyses:

Carbon	0.15	to	0.45	per cent
Manganese	0.20	"	0.40	"
Silicon	0.15	"	0.30	"
Chromium	12.0	"	14.0	"
Nickel	nil	"	0.5	"

This material is distinctly air-hardening, but possesses high physical properties. It can be hardened and tempered to give desired results, within the limits of the material, but these same air-hardening tendencies considerably restrict the easy fabrication of many articles. A material of this type, so far as hot working is concerned, should be fabricated within a range of temperature from 2100 to 1700° F. It will readily be seen that various delicate shapes and sections and such articles as light gage sheets are extremely difficult to finish without repeated reheatings in this temperature range, and in many cases it is almost impossible to finish at all above 1700° F.

The physical properties of this material are fairly well illustrated by the following tables taken from a pamphlet edited by Mr. Harry Brearley, of the Brown Bayley Steel Works, of Sheffield, England:

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[1] B. Strauss, "Non-Rusting Chromium-Nickel Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 208 (1924).

[2] F. A. Fahrenwald, "Some Principles Underlying the Successful Use of Metals at High Temperatures," *Ibid.*, p. 310.

[3] J. B. Johnson and S. A. Christiansen, "Characteristics of Material for Valves Operating at High Temperatures," *Ibid.*, p. 383.

STAINLESS IRON

Composition: Carbon 0.07 per cent; manganese 0.12 per cent; silicon 0.08 per cent; chromium 11.7 per cent; nickel 0.57 per cent.

Treatment: Oil-hardened from 930° C. and reheated as shown.

Reheating Temperature	200° C.	300° C.	400° C.	500° C.	600° C.	700° C.	750° C.
Yield Point, tons per sq. in.	....	....	....	58.8	38.0	30.6	27.9
Maximum Stress, tons per sq. in.	73.0	72.4	72.3	72.4	49.1	40.4	36.4
Elongation, per cent	12.0	12.5	15.5	18.0	22.0	26.5	31.0
Reduction of Area, per cent	38.0	36.4	51.0	52.2	62.4	65.8	68.8
Izod Impact Figure	34	38	38	36	65	79	87
Brinell Number	340	332	332	340	241	196	179

MILD STAINLESS STEEL

Composition: Carbon 0.15 per cent; manganese 0.16 per cent; silicon 0.09 per cent; chromium 11.8 per cent; nickel 0.77 per cent.

Treatment: Oil-hardened from 930° C. and reheated as shown.

Reheating Temperature	500° C.	600° C.	700° C.	750° C.
Yield Point, tons per sq. in.	....	42.0	38.0	31.2
Maximum Stress, tons per sq. in.	89.5	56.4	46.8	43.9
Elongation, per cent	10.0	20.0	26.0	28.0
Reduction of Area, per cent	36.0	52.2	58.1	61.5
Izod Impact Figure	16	35	60	68
Brinell Number	402	255	223	207

STAINLESS STEEL

Composition: Carbon 0.37 per cent; manganese 0.15 per cent; silicon 0.19 per cent; chromium 12.0 per cent; nickel 0.55 per cent.

Treatment: Air-hardened from 930° C. and reheated as shown.

Reheating Temperature	500° C.	600° C.	700° C.
Yield Point, tons per sq. in.	....	56.0	46.8
Maximum Stress, tons per sq. in.	104	63.0	54.0
Elongation, per cent	9.0	15.0	21.0
Reduction of Area, per cent	24.6	42.0	52.2
Izod Impact Figure	8	15	30
Brinell Number	444	285	241

Previous papers by metallurgists have given detailed information concerning the corrosive effect of this type of material in its various conditions,[1] [2] [3] [4] [5] so that the author leaves this matter to the reader with a statement that the type of material referred to above is in its best corrosive-resistant condition in the hardened state, that is to say hardened in such a manner and from such a temperature that the carbides within the mass are thoroughly diffused, giving microscopically a homogeneous structure. Any further drawing which would lead to dissolution, thus introducing lack of homogeneity, renders the material less resistant to corrosive attack.

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[1] Symposium on Corrosion-Resistant, Heat-Resitant and Electrical-Resistance Alloys, ***Proceedings***, Am. Soc. Testing Mats., Vol. 24, Part II, p. 189 (1924).

[2] J. H. G. Moneypenny, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., February, 1924.

[3] R. J. Anderson and G. M. Enos, "Corrosion-Resistant Alloys for Use in Acid Mine Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 259 (1924).

[4] Jerome Strauss and J. W. Talley, "Stainless Steels: Their Heat Treatment and Resistance to Sea-Water Corrosion," *Ibid.*, p. 217.

[5] Donald G. Clark, Am. Iron and Steel Inst., October, 1925.

2. *Chromium Alloys:*

In the second class there are well established types of steel at present on the market. One is of approximately the following analysis:

Carbon	under 0.10 per cent
Manganese	as desired 0.20 to 0.60 per cent
Silicon	according to requirements up to say 1.50 per cent
Chromium	16 to 20 per cent
Nickel	nil

This material is now classified as a "stainless iron," containing as it does over 16 per cent of chromium with extremely low carbon and substantial additions of silicon. This alloy has a hot working range of from 2100° F. down to "black heat." It shows no tendencies whatever to air-harden though the physical properties of the finished hot worked material are strictly correlative to the amount of physical work put upon it in fabrication.

In other words, a 1-in. round bar rolled from a suitable billet and finished in the neighborhood of say 1400° F. would have higher physical properties than a 3-in. round bar from the same size billet.

A peculiarity of this material is that the Brinell, Rockwell, or scleroscope hardness is barely affected by quenching the material at any temperature up to 1750° F., though the crystallin structure undergoes considerable change by prolonged heating at high temperatures. The highest Brinell on this type of material that the author has ever been able to obtain was on a $\frac{1}{4}$-in. thick plate rolled from a billet 30 by 9 by 50 in., at the Coatesville plant of the Bethlehem Steel Co., and finished at a temperature of approximately 1200° F. In this condition the material was readily sheared on both the straight type of shear, and the rotary shear. The physical properties of this plate were as follows, the chemical composition being: carbon 0.07 per cent, manganese 0.70 per cent, phosphorus 0.031 per cent, sulfur 0.010 per cent, silicon 0.64 per cent, chromium 17.04 per cent:

TREATMENT	ELASTIC LIMIT, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION IN 2 IN., PER CENT	REDUCTION OF AREA, PER CENT	BRINELL HARDNESS
As rolled................	75 040	105 020	8.00	14.00	235
Heated to 1550° F. held 15 minutes, cooled in air...	50 140	71 700	34.00	64.80	170
Heated to 1500° F., held 1 hour, cooled in furnace, piece heated up with furnace..............	48 560	71 200	33.00	60.90	187

Another steel in this group which is finding a substantial market comes within the following approximate analysis:

Carbon..................................	0.12	per cent
Manganese..............................	normal	
Silicon..................................	0.50	"
Chromium...............................	20 – 30	"
Nickel...................................	under 0.50	"

This material with its high chromium content does not harden materially by heat treatment, and its physical properties vary considerably according to the manufacturing conditions. It is claimed[1] for this type of material that the higher chromium content gives considerably increased resistance to oxidation at high temperatures, and increased resistance to certain corrosive media. So far as the author is able to state from actual experience, however, there are but few cases where the higher chromium material has shown any advantages over the material containing say 16 per cent chromium with silicon; whereas, it is now generally conceded that the chromium-silicon iron is much more easy of fabrication and has much less tendency to become brittle under heavy mechanical work, or to exhibit a seriously laminated structure so prevalent in a heavily worked 25 to 30-per-cent chromium iron.

The merits of these two materials provide an excellent subject for debate, but the author as stated knows of very few instances where the higher-chromium material (25 to 30 per cent chromium) has been found more advantageous in use than the somewhat lower-chromium alloys with a suitable silicon addition.

A distinct feature of the materials containing upwards of 16 per cent of chromium is that they are in many cases immune to corrosion due to contact with other metals (particularly brass and bronze). Where non-ferrous materials are used in conjunction with chromium alloys a material containing upwards of 15 per cent of chromium is advisable. Many of the lower chromium steels have suffered severely from this type of selective corrosion, particularly under salt water conditions.

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[1] C. E. MacQuigg, "Some Engineering Applications of High-Chromium-Iron Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 373 (1924).

3. *Nickel-Chromium Alloys:*

In the third class are to be found a series of nickel-chromium alloys covering a wide range in chemical analysis. Among these are some extremely useful materials, and it is to be regretted that since the introduction of the straight iron-chromium, or iron-chromium-silicon alloys, the nickel-chromium alloys have more or less been relegated into the background. This is possibly due to some of the difficulties encountered in fabrication and also possibly to the fact that the nickel-chromium alloys are naturally more expensive than the nickel-free alloys.

In the corrosive field two typical approximate analyses of the most prominent materials are given below, with the corresponding physical properties:

Carbon	0.30 to 0.50	per cent
Manganese	0.50 to 0.70	"
Silicon	1.0 to 1.50	"
Chromium	6.0 to 9.0	"
Nickel	22 to 25	"
Copper	1.0 to 1.5	"

Tensile Strength, lb. per sq. in.	105 000 to 125 000
Yield Point, lb. per sq. in.	75 000 to 95 000
Elongation, per cent	25 to 35
Reduction of Area, per cent	40 to 55

Carbon	0.30 to 0.50	per cent
Manganese	0.50 to 1.00	"
Silicon	0.50 to 1.00	"
Chromium	12 to 15	"
Nickel	20 to 30	"

Tensile Strength, lb. per sq. in.	100 000 to 150 000
Yield Point, lb. per sq. in.	80 000 to 110 000
Elongation in 2 in., per cent	20 to 35
Reduction of Area, per cent	30 to 50
Brinell Hardness	225 to 300

This type of material is usually austenitic, and therefore physical properties are somewhat restricted so far as heat treatment is concerned. Moreover, in the original cast ingot form the material is somewhat fragile and uncertain, and difficulty has been experienced in its satisfactory manipulation.

Within the last few months a material of the following approximate analysis has come to the front as corrosion-resistant material, and it has many excellent features:

Carbon	0.10 to 0.20	per cent
Manganese	0.20 to 0.40	"
Silicon	0.30 to 0.60	"
Chromium	16.00 to 18.00	"
Nickel	6.00 to 9.00	"

It has not, however, so far been developed to any marked extent in this country, although it has gained a certain amount of popularity in Europe.

Before passing on to the fabrication problems I wish to say a word about the conditions and duration of corrosion tests, which have led to many peculiar results. For instance, many straight solutions of acid in water give entirely different results from those obtained with complex, natural, or built up solutions with the same acid content. For example, low percentages of sulfuric acid in water will seriously attack rustless irons and steels, but often in mixed solutions such as mine water there is no attack. Again, short duration tests under laboratory conditions cannot always be taken as indicative of longer service. The author has seen material show the usual slight loss under the first few days of test, and then remain perfectly stationary for as long as 120 days, only to collapse rapidly. He has seen material treated in nitric acid solution withstand sulfuric acid for periods of as long as 30 days without loss of weight. These short duration tests can be very misleading at times and these remarks are made to sound a note of warning to some of our over-enthusiastic chemists and metallurgists. For mixed acid conditions the author believes a suitable high-nickel high-chromium alloy[1] will ultimately be adopted and it behooves the manufacturers of such material to get into sheet and structural shape production promptly.

In this paper the author has particularly avoided references to patents and may have left out many deserving materials, which, however, he feels will come within the three classes mentioned. The typical analyses given have been used as indicative of such classes rather than intended to apply to any particular brand of material or trade name.

FABRICATION OF CORROSION-RESISTANT ALLOYS

The intention of this paper is to deal rather with the practical fabrication than with any of the purely technical problems. For several years those interested in the production of corrosion-resistant materials have been engaged in expensive experimental and demonstrative research work in fabrication problems to establish limits and claims for their various products. This new industry has waited for the establishment of the confidence of the user. Within the last few months a new incentive has been given to this work when one of the largest explosive chemical manufacturing concerns in the world issued an order for a series of tanks to be used in a process for the manufacture and storage of nitric acid. An order of such magnitude brought into the structural field many problems which neither the scientist nor the chemist had foreseen: problems which themselves narrowed down the choice of materials by the very demands made upon them. The building of this installation involves the production of sheets, bars, rivets, angles, channels, beams, shapes, seamless tubes, flanged and dished heads of unusually large size, and castings, with the satisfactory assembling of these parts into a complete unit capable of meeting definite specifications with regard to physical properties, pressure tests, and corrosion resistance. With the placing of this order the designing engineers, as well as the practical man, who previously had been prone to regard all so-called rustless or stainless irons and steels as very similar, found such to be far from the case.

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[1] W. H. Hatfield, *Engineering*, November 20, 1925.

The chemist had eliminated certain steels, which although rustless and stainless in the ordinary sense of the word were not satisfactory so far as attack by nitric acid was concerned. Other types of material, although resistant to nitric acid had to be abandoned either on account of prohibitive prices or the fact that they were not available in some of the forms necessary for the building up of this equipment.

In submitting a suitable material for this particular service, it was essential to keep in mind the two fundamental[1] principles of corrosion, that is, (1) solubility; and (2) possibility of electrolytic action where two dissimilar materials were used together. With these problems confronting them the engineers found that despite fourteen years of hard work by numerous steel manufacturers on the subject of corrosion resistance, they had out of all the accumulated data to find a material:

1. Resistant to the nitric acid conditions;
2. Possessing the physical properties desired;
3. Capable of being produced in the various shapes, sections, etc., representing the component parts of the unit; and
4. Capable of satisfactory assembling into the finished unit.

The specifications called for material having the following physical properties:

Tensile strength, lb. per sq. in.	60 000
Yield point, lb. per sq. in.	40 000
Elongation in 2 in., per cent	25
Reduction of area, per cent	50
Bend Test	120 deg. without fracture.

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[1] W. D. Bancroft, Am. Electrochemical Soc., October, 1924.

The engineers knew as did the steel manufacturer that these specifications can readily be met by many corrosion-resistant steels. The specifications may ultimately be tightened up somewhat, but every assistance was given the manufacturer in order to secure his full cooperation. Up to this point the choice of material can safely be left to the engineer, chemist and metallurgist, but from now on the practical man and his operations must be considered.

In the form of large sheets there are available on a production basis only the chromium-silicon-iron alloys, straight chromium iron alloys and the 25 to 30-per-cent chromium low-carbon alloys. Sheets required for the fabrication of tanks must be capable of a considerable amount of cold work and must of necessity be capable of being drilled, or punched if possible.

The manufacture of rivets presented probably the greatest problem of all, for whereas it is possible to produce an ideal rivet from the rivet makers' standpoint from a fairly wide choice of materials, the reheating of the rivet for driving considerably narrowed down the choice of materials. Disastrous results were experienced in the early stages of experiment with materials that had distinctly air-hardening tendencies, for as rivets of such materials cooled down they automatically became hardened, and the heads would be quite brittle, so that if they did not actually fly off under tension they could be knocked off with the slightest blow or jar. It therefore became clear that non-air-hardening material would be absolutely essential for this purpose. The hot working of this material again brought up this question, for whereas air-hardening material had to be abandoned, the chromium-silicon irons and the higher chromium materials had to be manipulated in such a manner as to avoid undue grain growth, which is a feature of all these materials.

So far as the author is aware no angles or channels such as called for in this equipment had ever been produced commercially before. Chrome-silicon iron was selected for this particular purpose, because of the working range of temperature necessary, the heavy drafts involved, and the mobility of this material at reasonably low temperatures.

In the manufacture of seamless tubing, so far it has been found practicable to pierce in a commercial manner only chromium-silicon iron and the low-carbon high-chromium irons, but to-day there are available other forms of tubing of the welded type which for many purposes are quite satisfactory. However, as there is often a great tendency for welded tubes to fail under acid conditions seamless tubes were insisted upon in this particular case. Such tubes are now available in sizes from 6 in. down to 2 in. and in cold-drawn form in some of the smaller sizes, though in the smaller diameters and thinner walls the industry has not progressed as far as in the case of the larger diameter with wall thicknesses of say No. 9 gage and heavier.

The building of the series of tanks referred to called for single piece heads and bottoms, and these in turn required plates varying from 90 to 145 in. in width and in $\frac{1}{4}$, $\frac{5}{16}$ to $\frac{3}{8}$ in. thicknesses. So far as the author is aware no plates approaching 145 in. in width had previously been rolled, but this was successfully accomplished at the plant of the Worth Steel Co., Claymont, Del.

Those acquainted with the flanging and dishing operation will readily appreciate the difficulties of handling a highly-alloyed iron under such conditions. In dishing the tendency of the material to corrugate or buckle presents considerable trouble. Further, the forming of the flange, in which the rim only of the material is subjected to mechanical deformation and is cooled in many cases at an entirely different rate of speed than is the body of the plate, at almost "black heat" calls for a material free from the slightest tendency to air-harden, and capable of holding within itself, without causing rupture, the strains set up in the dishing and flanging operations. The author has seen both low and high-chromium material (chromium 25 to 30 per cent) formed and almost finished, go simply to pieces in the actual finishing, or even sometimes hours after finishing, due to strains within the material set up by the uneven heating conditions which prevail in this particular operation.

From the above description it should be obvious that the practical man is now reaching that point, hitherto untouched by the chemist and metallurgist, where his various operations which cannot fundamentally be altered become often the deciding factor in the choice of a suitable material, if not at first then assuredly later, in many cases only after costly experiments and serious failures.

The selection of the iron-chromium-silicon alloy for this particular problem was made only after the following conditions had been filled:

1. The chemist had been satisfied of the resistance of the material to the acid condition;

2. The metallurgist had established that the chemical and physical specifications could be met;

3. The practical man—"the man at the fire," so to speak—had been satisfied by actual demonstration that his standard practices, with but slight modifications in some cases, were quite capable of handling the type of material.

Plates.—As emphasizing the latter point, it was found that plates could be rolled from suitable billets over a temperature range of from 2100 to 1400° F., with standard drafts and without undue trouble. Rolling too cold resulted in a wavy plate, which could not readily be straightened due to the fact that the tensile strength and elastic limit are both increased by cold working. The finished plates could be sheared either on straight or rotary machines without intermediate annealing.

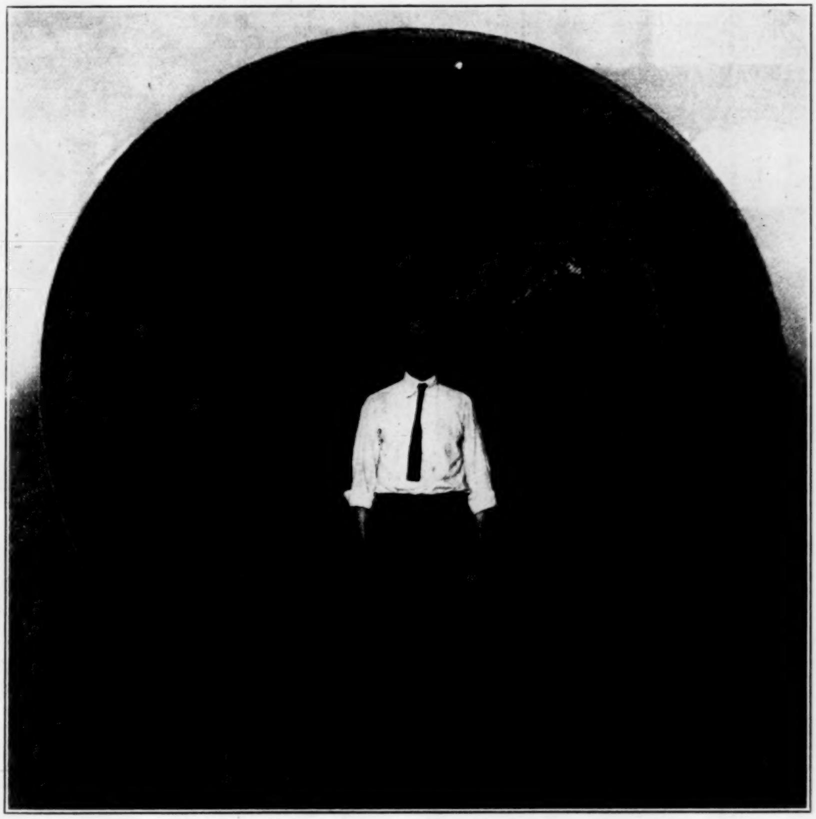
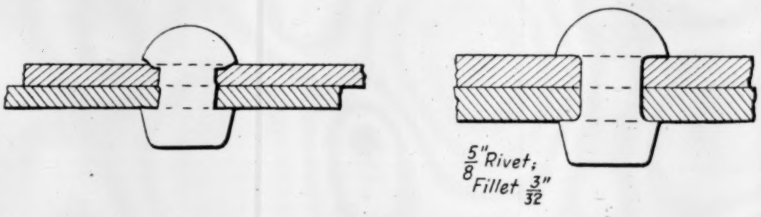


FIG. 1.—Showing Flanged and Dished Head.

Flanged and Dished Heads.—These can be produced from this material by almost standard process, though the operations are prolonged by the necessity of reheating more frequently, due first to the increase of the tensile strength and elastic limit by cold working, rendering forming more difficult, and secondly to the fact that it is inadvisable to use too high a temperature on the finished plate, only the outer rim of which is submitted to mechanical work. Such a condition would produce an undesirable difference in grain structure.

Rivets.—It has been established that hot, or cold made rivets of this alloy can be produced commercially. A word of warning, however, should be given. Few manufacturers so far have undertaken this work. The indiscriminate ordering of chromium-iron rivets would be disastrous and mean a reversion to some of the unfortunate and expensive results encountered in some of the early fabrication work. Practical work in this direction has established the following:

1. A satisfactory bar from which to commence manufacture is essential. All material should be capable of withstanding a 90 to 120-deg. bend.



(*a*) Rivet without fillet; no countersink.

(*b*) Showing slightly countersunk hole, rivet filleted.

FIG. 2.—Rivet Driven for Test Purposes, $\frac{5}{8}$ in. in Diameter, $\frac{11}{16}$-in. Hole.

2. The rivet head must be formed within such a temperature range that the physical properties of the original bar are not impaired. This is not the easiest condition to fulfill inasmuch as the formed head is the only portion of the rivet which receives any mechanical work. An ideal head with an overheated, or coarsely crystallin shank is useless. The forming of the second head in fabrication would probably produce a satisfactory head, but the junction between the heads and shank would be weak. Such heads will fly off under a pressure test.

A close study of the rivet question brought to light a peculiar condition. In all high-grade tank production the holes in the shell are reamed to size. The rivet maker uses a bar varying in size from 0.01 to $\frac{1}{64}$ in. bare, for two reasons: the easier clearance in his automatic machines, and the fact that the hot rivet is easier to place in the shell. This produces the condition illustrated in Fig. 2 (*a*). It will be noted that the barrel of the rivet "swells" and forms a perfectly tight fit in the hole, but the swelling of the barrel and the forming of the head leaves a distinct V notch where the head joins the barrel. Since a V notch represents the weakest possible condition so far as shock or shear load is concerned, it is suggested that a rivet reasonably "filleted" at this point is much superior. A countersunk hole and substantially "filleted" rivet as illustrated in Fig. 2 (*b*) would undoubtedly be the best, but cost and difficulty of manufacture are facts that must be considered.

Angles and Channels.—Beyond extra care in heating, due to the poor conductivity of the material, no unusual conditions are necessary in the production of these special sections. So far as the author is aware these sections were the first produced in commercial quantities in the United States.

Castings.—The production of castings capable of being riveted to a tank shell presented problems which had previously caused difficulties. A casting with suitable chromium content but too low in carbon exhibited the usual crystallin structure of ferro-chrome, although the cooling effect of the casting also had some bearing on this. Such a structure is often associated with porosity. Material of the following approximate analysis has so far proved satisfactory and after suitable heat treatment has been riveted successfully to tanks and in instances has been calked without the use of calking strips, though this is not advocated as a general practice:

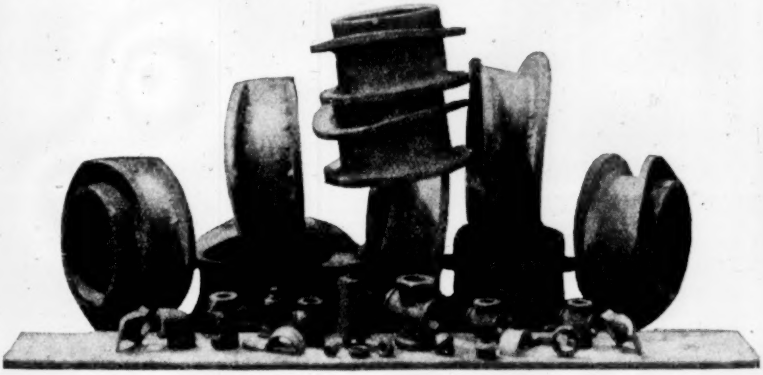


FIG. 3.—Castings of Chromium Steel.

Carbon	0.20 to 0.40	per cent
Silicon	0.60 to 1.00	"
Manganese	0.30 to .60	"
Chromium	16.50 to 18.50	"
Nickel	under 0.25	"

Final Assembly.—The final building up of the unit is the real test of a suitable material. It is here, with the bulk of the labor and

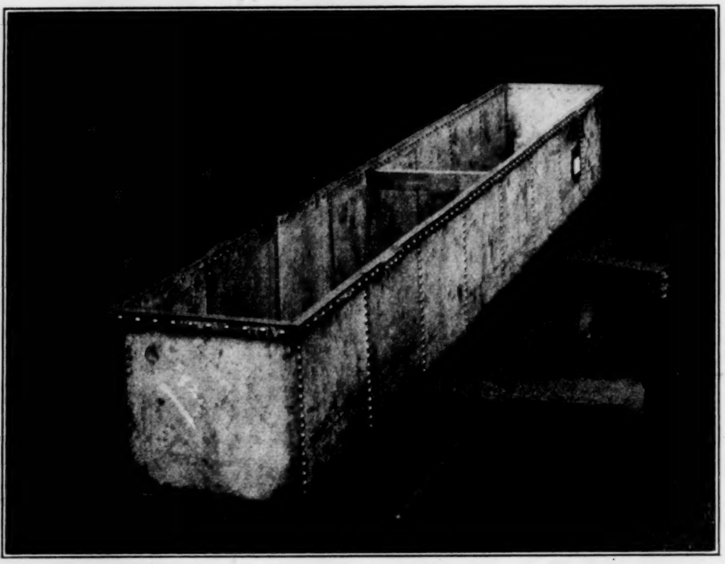


FIG. 4.—Chromium Steel Tank.

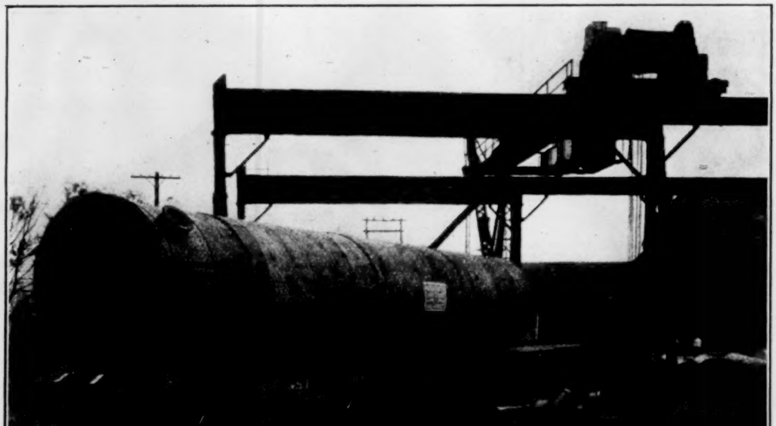


FIG. 5.—Chromium Steel Tank.

expense locked up in material form, that the wise, or unwise choice of material is evidenced. The author has known several months of labor and effort in tank construction to be entirely lost due to lack of knowledge.

Most of the chromium alloys, stainless steel, rustless irons, etc. —call them what you will—can be put in a condition so that they will form in bending rolls, or stand punching or drilling as the case may be. Laminated plates, however, often split or "spawl" during calking. Heads on unsuitable rivets will be "weak" or fly off under test. Unsuitable castings will crack with the local application of a hot rivet and under the blows of a riveting hammer, as will plates that have acquired a certain amount of mechanical hardness in the manufacture. Despite these difficulties, satisfactory tanks can be produced from a suitable material, as illustrated in Figs. 4 and 5.

The Downingtown Iron Works are now engaged in the production of tanks from this material. Some of these are 10 ft. in diameter and about 50 ft. long or high and others 17 ft. long.

In conclusion, the author wishes to say that he has endeavored to point out that there is a field for all of these rustless, or stainless materials and he has cited the equipment described merely to bring home the fact that often the actual fabrication operations, or the physical properties required are the deciding factors in the final selection of the material. He urges the steel manufacturer to be conservative in his claims for his particular product and the user not to expect from this highly alloyed material quite the same ease in fabrication as with ordinary mild steel. Caution on the one hand with cooperation on the other will lead to the solution of many problems hitherto considered insurmountable.

As a concluding remark it would seem from the author's work that an alloy containing very low carbon with upwards of 16 per cent of chromium is a decided advantage in work of this nature as such materials are more readily fabricated and more resistant to corrosion than the somewhat lower chromium ranges.

Acknowledgement.—The author takes this opportunity of expressing his thanks to the following companies for their painstaking efforts in assisting him to obtain results:

1. Messrs. E. I. DuPont de Nemours and Co.;
2. Bethlehem Steel Co., Coatesville Plant;
3. The Downingtown Iron Works;
4. Phoenix Iron Works Co.;
5. The Worth Steel Co.;
6. Alan Wood Iron and Steel Co.

DISCUSSION

Mr. Dixon. Mr. E. S. Dixon.[1]—I should like to ask Mr. Nelson if he has any data on the ability to weld these chromium steels, either electrically or with acetylene.

Mr. Nelson. Mr. T. H. Nelson.—Yes, we can weld them; but unfortunately, few of us can defeat the laws of nature and so far we have not been able to prevent the crystallin growths of this alloy. We can get a liquid-tight or an air-tight weld, but we cannot get a very tough weld; the best I know of is to make the welding operation as rapid as possible and introduce any method we can to prevent grain growth. Experiments I have recently conducted, however, lead me to believe that it may be possible to retain a substantial degree of toughness even with fairly large crystallin growth.

Mr. Bain. Mr. E. C. Bain.[2]—Will Mr. Nelson tell us more about the effect of increased silicon content in rustless iron to resist corrosion when in contact with non-ferrous metals? And further, I should like to ask Mr. Nelson if he has at hand information which he will impart on brittleness. Is the brittleness mentioned always the result either of the self-hardening effect or of excessive grain growth, or are there still other causes of brittleness to be found?

Mr. Nelson. Mr. Nelson.—With regard to the first point, contact corrosion with non-ferrous metals, I have felt that at a later date such a subject is worthy of a special paper, and therefore I only touched lightly on it at the moment. With over 16 per cent of chromium I do not find the addition of silicon essential. It is, however, undoubtedly an advantage; 18 per cent chromium and 0.75 to 1.50 per cent silicon may be used with satisfaction. On the other hand I think 25 per cent chromium, possibly without silicon, would have a similar result. It is essential that upwards of 15 to 16 per cent of chromium be present to get away from contact corrosion with non-ferrous metals.

As to the second point, the question of brittleness travels the entire range of iron-chromium alloys and is not altogether tied up with grain growth, neither is it tied up with chromium content. The 16-per-cent chromium limit seems to be a serious feature in more ways than one. Not only is it the demarcation line between the material that will and the material that will not harden, but it seems to be a saturation point with iron in chromium which coincides with many things, contact corrosion, for one, resistance to shock for another. **Mr. Nelson.**

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[1] Metallurgist, The Texas Co., Port Arthur, Tex.

[2] Metallurgist, Union Carbide and Carbon Research Laboratories, Inc., Long Island City, N. Y.

MR. H. C. KNERR.[1]—A significant development in the use of these alloys is in structural parts of aircraft, especially sea planes, where highly stressed parts made from sheet or tubing may, for lightness, be as thin as 0.035 in. A small amount of corrosion evidently would cause serious weakening. Certain of these alloys may be acetylene welded with excellent results. Some may be severely cold formed and have been drawn in seamless tube down to the size used in hypodermic needles. **Mr. Knerr.**

MR. F. N. SPELLER.[2]—I presume that Mr. Nelson refers to 18-per-cent chromium steel in the samples of tubing he speaks of and that he refers to the fusion welding processes. Our experience in the manufacture of seamless tubing has so far been in favor of a chromium content of over 20 per cent. Paradoxically, the difficulty seems to be to make a seamless tube of this steel free from seams. The inspection of such material is very close, and one seam in the middle of a 20-ft. tube will often cause the loss of that length, and the scrap has no great value. These are practical considerations that have had much to do with the economic production of chromium-iron seamless tubing. **Mr. Speller.**

MR. NELSON.—The question of seams is not confined to chromium iron; it is a trouble of an entire industry, and the cure at one end of the industry is invariably the cure at the other, and goes back to the steel maker, or some of the processes. The piercing operation in this material itself is conducive to leaving slight laminations, scoring, or fissures inside, because the resistance of this material, even at temperatures considerably elevated, is so much greater than that of mild steel, and the material therefore does not conform any too readily to piercing conditions. Sources of supply are very limited, but both seamless and welded tubes are available. **Mr. Nelson.**

MR. J. H. PARKER.[3]—I quite agree with Mr. Nelson that the field of application of various percentages of chromium in stainless steels deserves a lot of study. There is the 14-per-cent chromium type; there is the type just over 16 per cent; there is the 20-per-cent type, and on higher. It takes a careful study of the individual problem to find out just which one is going to best fit in. I am not in accord with Mr. Nelson when he thinks that chromium over 16 per cent constitutes the cure-all of this iron-chromium situation. Chromiums **Mr. Parker.**

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[1] Consulting Metallurgical Engineer, Philadelphia, Pa.

[2] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

[3] Vice-President, Carpenter Steel Co., Reading, Pa.

Mr. Parker. under 16 per cent seem to have no friends in this discussion, and I want to say a word for them.

Tubing, both hot-rolled and cold-drawn, has been made from stainless iron of from 12 to 14 per cent of chromium. I have a sample of cold-drawn tubing with me, and would be glad to show it to anybody that would like to see it. It is made out of 12.50-per-cent chromium steel. This same percentage chromium stainless iron has been worked into a variety of hot-rolled sizes.

In the matter of toughness, 12 to 14-per-cent chromium alloy has a big advantage over the higher chromium alloys. Another point in its favor is that in oxy-acetylene welding there is much less grain growth than in the higher chromium steels, and while the welding will become somewhat hard, this can be taken care of by a blow-torch anneal at the weld. These low-chromium stainless irons will also withstand a higher forging temperature without grain growth than will the higher chromium steels.

Mr. Nelson. MR. NELSON.—I wish to reply to Mr. Parker that I do not appear here to-day in the light of a particular friend of alloys of over 16 per cent chromium. My early association with Mr. Harry Brearley, the discoverer of the 12 to 14-per-cent chromium alloy, is such that I fully appreciate not only the man but the material.

The operations in which I have been engaged for the last five or six months, due to the various processes involved, have of necessity called for a material that exhibits no air-hardening tendency whatever. This paper, therefore, dealing with that subject only, refers particularly to the higher chromium alloys as applicable to that job. I frankly stated in the paper that there are many instances where I would confidently advocate the use of lower chromium materials.

I should like to ask Mr. Parker if he will tell me the tonnage of 12 to 14-per-cent chromium tubes that have been drawn and are now available, and whether they were made in this country.

Mr. Parker. MR. PARKER.—The tubing, both hot-rolled and cold-drawn, was made in this country. The cold-drawn tubing is, however, a comparatively recent development. There have been at least 15 to 20 tons of hot-rolled tubing made during the last ten months. Of the cold-drawn a dozen bars have recently been produced without difficulty, since the first rolling went through in good shape and there is no question about its practicability.

Mr. Mochel. MR. N. L. MOCHEL.[1]—This is not in criticism of what Mr. Parker has just said, but I want to say, in behalf of Mr. Nelson, that he is not

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[1] Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., Lester Station, Philadelphia, Pa.

Mr. Mochel. a firm advocate of the alloys over 16 per cent chromium. The patent restrictions have been such that they have held up the development of these promising alloys to a great extent. Many manufacturers have had their hands tied and have not been able to cover the entire field. Several have not had open minds on the subject, but I have always found the contrary to be true with Mr. Nelson. He has had the courage to say "no" when he knew his particular alloy would not serve best for the purpose.

Mr. Nelson has stressed in his paper the necessity of carefully considering every aspect of a problem before going into the selection and use of any of these alloys. There is no one alloy now available that will serve every purpose. Personally, I have found it more or less necessary to use the 11.5 to 13-per-cent chromium alloy for certain purposes; and where high elastic limit or high physical properties in general are necessary, one must needs go to the lower chromium alloys, especially with the low-carbon or "iron" grades. If one does not care especially regarding physical properties but is more concerned with resistance to corrosion, he will naturally go to the higher chromium alloys, often to those with high silicon or copper as well. The physical properties, the degree of corrosion resistance, the nature of the corroding medium, the method of fabrication, must be equally considered. The last is a most serious matter, as a given alloy may satisfy the first conditions, and yet cannot be fabricated economically or safely for the purpose intended. Fabrication is the most important factor from a cost standpoint.

Mr. Nelson has also dealt briefly with the nickel-chromium rustless alloys. It is gratifying indeed to find one whose name has been generally linked with the development of straight chromium alloys, advocating the consideration of the nickel-chromium types. Many have tried to pass over them lightly and claim there was no advantage to be derived from their use, and yet the addition of but 1.50 to 4 per cent of nickel will give surprising results in alloys of 11.5 to 14 per cent of chromium.

Mr. St. John. MR. ANCEL ST. JOHN.[1]—Mr. Mochel has referred to a point that Mr. Nelson brought out which I want to emphasize. I have no personal interest in the manufacture or the use of these alloys, but I do have a very great interest in the general field of proper selection and use of material, and I am persuaded that this patent situation and the unenlightened activities of sales departments have been very dangerous. I speak with a certain amount of knowledge with respect to that, because of things that have been brought to

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[1] Consulting Physicist, New York City.

Mr. St. John. my attention. For instance, a bar of chromium iron alloy to be used as the spindle in a high-pressure valve was being examined by a group of prominent metallurgists and a consulting engineer. Unfortunately somebody's hand slipped, the bar dropped on the floor and was no longer a bar but a series of fragments. What is the impression created in the minds of consulting engineers who are not particularly versed in metallurgy themselves, mechanical engineers, if you please, when they see a material that has been recommended to them for spindles in high-pressure valves go to pieces when they happen to let go of it? It seems to me that the use of chromium-iron alloys gets quite a jolt there. As a matter of fact, I have been told that one of the men who saw that said, "No more of that material for me; I am going to change my specifications right now."

It seems to me that incidents of this kind portray a serious situation, one which should be avoided in the future by intelligent cooperation and intelligent salesmanship, if you please. The people who have alloys to sell are doing themselves, as well as the entire industry, a permanent injury by trying to sell a customer a material which he has no business to buy.

Mr. Mathews. THE CHAIRMAN (MR. JOHN A. MATHEWS).[1]—I think the hour has arrived when we must close this discussion. I should like to add to what has been said, that Mr. Nelson has shown a very open mind in reference to the various types. He has been at it long enough to know their merits and shortcomings, and most of us who have experience in these fields, including the chromium-nickel alloys that have been touched on lightly to-day, know that that is true. The most difficult problem in connection with the use of non-corrosive steels is in selecting the right type for each application, for this includes not only corrosion resistance but a knowledge of mechanical and thermal properties; their relation to other metals used in the same construction, and fabricating qualities as well.

Will you close the discussion, Mr. Nelson?

Mr. Nelson. MR. NELSON.—I wish to thank those who have entered into the discussion, for their kindly criticisms, and in conclusion I should like to refer briefly to Mr. St. John's remarks and then go back to Mr. Parker's comments for a moment. We are confronted many times with high-temperature superheating problems that demand certain physical properties at certain temperatures. At the temperature in question, in the incident cited by Mr. St. John, the material was very fragile, but it was proved that at the operating temperature involved the material was very tough, and that was a very valuable discovery.

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[1] Vice-President and Metallurgist, Crucible Steel Co. of America, New York City.

Mr. Nelson.

I give that as an instance that the operating conditions are often more truly representative of the facts than are standard specifications.

With reference to Mr. Parker's discussion, it comes to my mind that last week I had a paper from England which was read by Mr. Waddell, wherein he himself—and I take it, it is true of Mr. Brearley's laboratory and laboratories in general—advocates 15.5-per-cent chromium alloy for use in conjunction with non-ferrous alloys, to avoid contact corrosion, and I think that if Mr. Brearley were here in person he would exhibit a practical open and frank mind and say that there are virtues and vices in all.

As the 12 to 14-per-cent chromium steels can be hardened and tempered, the maximum degree of toughness obtainable follows the general principle of a hardened tempered steel. The materials, however, containing over 16 per cent of chromium with low carbon and silicon, which do not harden, seem to possess maximum toughness when suitably worked mechanically, either in the forge or mill, and then drawn to approximately 1200 to 1400° F.

There is a brittleness found in the higher chromium series, 16-per-cent chromium, and up, which is more or less a phenomenon. I am working on this at the present time, but do not believe it is due to the air-hardening effect nor yet entirely to grain growth. I think the answer will be found in the condition of the solution of carbides at the starting point of manufacture, that is, in the billet form, and how ultimate mechanical work may impede or aid their dissolution. Tempering after mechanical work on over 16-per-cent chromium steels gives at present very varied results, but I am hoping by more careful study of the above to find the solution and explain at a later date this phenomenon of brittleness.

Mr. Strauss.

MR. JEROME STRAUSS[1] (*by letter*).—The presentation of facts concerning the processes necessary to the successful production of commercial fabricated forms in the corrosion-resistant alloys is indeed welcome, and the greater detail in which it is considered, the more valuable will it be to those who desire to manufacture equipment with these materials and to those who desire to extend the application of their products.

The writer is in close agreement with Mr. Nelson on his figures for suitable forging temperatures for the higher-carbon chromium steels and also the high-nickel-chromium steels; in the case of large sections, however, forging cannot be carried in the case of the higher-chromium low-carbon steels to as low a temperature as can be suc-

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[1] Material Engineer, U. S. Naval Gun Factory, Washington, D. C.

Mr. Strauss. cessfully practiced in small forgings and in sheets, at least not without disastrous results.

Mr. Nelson states that rivets of the low-carbon high-chromium alloys can be satisfactorily employed provided heating temperatures are not in excess of 1500° F. Although this is an improvement over the practice necessitated by the use of the low-carbon lower-chromium steels, it does not represent as great an advantage as might be imagined. The writer's experience has shown that satisfactory riveting may be accomplished with the various commercial low-carbon alloys at some temperature varying from 1400 to 1550° F., depending upon the actual type composition; this range is not particularly large. Successful attempts have been made to so modify the composition of the low-chromium material as to decrease or almost entirely inhibit hardening due to high temperature heating; these efforts have not thus far been entirely successful, for those elements which have been shown to inhibit hardening also promote grain growth at high temperatures. On the other hand, the high-nickel-chromium alloys are not subject to these defects and produce satisfactory rivets within a riveting temperature range of 1400 to 2050° F.

These same properties of the low-carbon chromium steels—air hardening of certain compositions and grain growth in other compositions—are of extreme interest in another operation common to the fabricating industries, namely, welding. On large structures the difficulties experienced in this work are not so readily overcome and it would be appreciated if Mr. Nelson could add to the available knowledge on this subject. More data concerning the properties of castings of these materials would also be of great interest, in particular the effects of heat treatment, data on tension tests, foundry properties, ability to secure pressure-tightness, etc.

In his reference to the higher-carbon chromium steels Mr. Nelson states that the least satisfactory condition to resist corrosion is the annealed state. The writer has shown[1] that for certain compositions, including some in the cutlery steel range, greater corrodibility is obtained at a temperature just beyond that which causes marked decrease in hardness, than is obtained on annealing. Since the preparation of that report, these facts have been checked on a number of occasions, thus affording proof of their reliability.

Mr. Nelson. MR. NELSON (*author's closure by letter*).—In reply to Mr. Jerome Strauss I should say that on the subject of rivets apparently I did not make myself quite clear. To my mind, the ideal rivet is the

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[1] Jerome Strauss and J. W. Talley, "Stainless Steels: Their Heat Treatment and Resistance to Sea-Water Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 246 (1924).

Mr. Nelson. low carbon, 16.50 to 18.50 per cent of chromium, with say 0.75 per cent of silicon—a cold-made rivet from the so-called annealed stock. Such a rivet eliminates many possible errors and abuses in heating so far as the manufacture of the rivet is concerned. This rivet can be driven at temperatures from 1500° F. down and the metal flows quite readily even at black heat, quite sufficient to swell the shank of the rivet and without inducing brittleness. This is quite an advantage over the lower chromium series without silicon.

I fully endorse Mr. Strauss' remarks regarding the nickel-chromium series, but there are instances of corrosion where the nickel-chromium materials do not stand, and there is also the desirability of using one material throughout when possible.

In summary, I should say that the nickel-chromium alloy has the widest range for hot riveting, the chromium-silicon alloy a range of at least 500° F., and the lower chromium alloy without silicon less than 200° F. I can see no objection to using nickel-chromium where corrosive conditions permitted and the price was not prohibitive.

With regard to large sections in hot working, such as forgings, etc., the author fully realizes that such difficulties have arisen, but these have been largely due to either lack of thought or an incomplete study of the subject. For instance a large forging, which is partly worked from time to time from every day forging heats, with the necessary reheatings, will exhibit large internal grain structures. The practice usually in such instances is to use a temperature of 1900 to 2000° F. The grain on the outer shell only is broken up by work and in intricate designs there are areas heated which receive say no work at all. If the standard practice is changed so that an original heat of 1900° F. was used only when considerable amount of work was necessary such as a general roughing down over an entire forging and then the local finishing work carried out at from 1400° F. down, I think results entirely superior to those intimated by Mr. Strauss would be obtained. I am glad, however, the point has been raised because it has been the problem of many. Large forgings, however, can be made without the undesirable large crystallin structure if care and thought are used in the manner of working down from the billet. A final heating to 1250° F. and cooling rapidly in air or even quenching has a decided toughening effect.

In conclusion may I thank all those who have entered into the discussion of this paper? I feel that such discussions are mutually beneficial.

THE MICROSTRUCTURE OF ZINC COATINGS[1]

By William H. Finkeldey[2]

Synopsis

This paper describes the preparation of specimens of galvanized materials for microscopical examination. Under this head is included the various methods of mounting specimens so they may be properly sectioned and polished, also the technique of etching which has been found to give the best results.

There is some discussion of the structural constituents which make up the coating, both from the standpoint of what one might expect from the iron-zinc diagram and also on the basis of what can be actually seen in the average microsection. The reader is cautioned against the too literal use of the terms $FeZn_3$ and $FeZn_7$ until more is known about the exact composition of the various iron-zinc constituents and a method developed which will permit their accurate identification under the microscope.

A number of photomicrographs are given illustrating the types of zinc coatings produced by the various hot galvanizing processes now in use in coating wire and sheet. The distortion, cracking and flaking, which such zinc coatings undergo when they are severely deformed, is also illustrated by photomicrographs. The various factors in the galvanizing process, which have an effect on the type of zinc coating produced, are enumerated. A few general observations on the relation of microstructure to the bending properties of zinc coatings are also given.

Photomicrographs are included to show the types of zinc coatings produced by the electrogalvanizing, sherardizing or sprayed zinc process.

The application of protective metallic zinc coatings to iron and steel has been practiced commercially for almost a century and the microstructure of metals has been studied for the past fifty years, but it is only within comparatively recent times that the microstructure of galvanized coatings has received much attention. Although Rawdon,[3] Raydt and Tammann,[4] Arthur and Walker,[5] Guertler[6] and others have all made valuable contributions to our fund of information on this subject, there still remains a large field to be explored.

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[1] A contribution from the Research Laboratories of the New Jersey Zinc Co.

[2] Assistant to Chief of Research, Research Division, New Jersey Zinc Co., Palmerton, Pa.

[3] H. S. Rawdon, "Structure of Commercial Zinc Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part I, p. 216 (1918).

[4] V. Raydt and G. Tammann, "Structure and Properties of Zinc-Iron Alloys Melted Under Pressure," *Zeitschrift für Anorganische Chemie*, Vol. 83, p. 257 (1913).

[5] W. Arthur and W. H. Walker, "Structure of Galvanized Iron," Am. Inst. of Metals, Vol. 6, p. 82 (1912).

[6] W. Guertler, "Structure of Galvanized Iron," *Zeitschrift für Metallographie*, Vol. 1, p. 353.

It is with the hope of making further additions to this general store of knowledge that the present paper has been written. However, in its preparation a number of points have arisen which cannot be satisfactorily explained. The far-reaching importance of these unanswered questions suggests that in this particular field of metallography our greatest advances are still to be made.

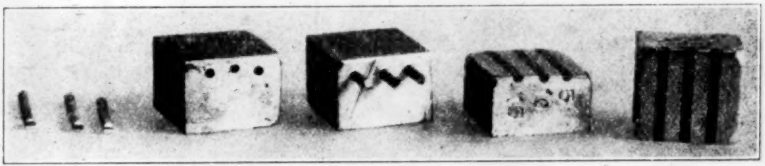

FIG. 1.—Soft Zinc Mount for Galvanized Wires.

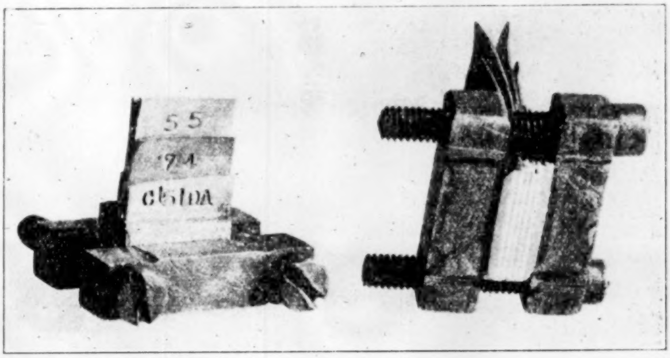

FIG. 2.—Galvanized Sheets Between Sheets of Soft Zinc Mounted in Clamp.

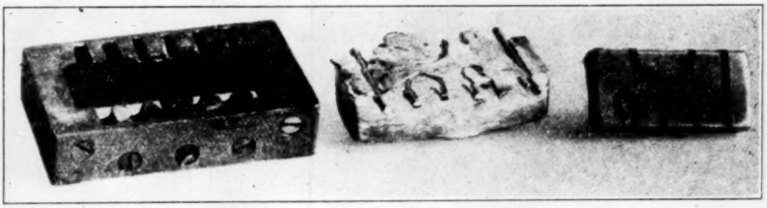

FIG. 3.—Mold Used in Casting Mounts With Molten Zinc. Mount Before and After Sectioning and Polishing.

PREPARATION OF MICROSECTIONS

In preparing samples of zinc-coated materials for examination under the microscope, it is absolutely essential that some form of mounting be used which will reinforce and protect the outside edge of the coating during the polishing operation. This can be accomplished in a number of ways; two of the best are illustrated in Figs. 1, 2 and 3. In one case, mounts of pure soft zinc in the form of rolled bars or thin sheets are slowly flowed into intimate contact with the surface of the zinc coating by pressure applied by a vise and clamps. In the other, the specimens are placed in a small iron mold secured in the proper position, and molten zinc, at a temperature just above the melting point, is poured around them.[1] The first method is not well adapted for the mounting of bent galvanized wires or sheets or objects of irregular shape; the second method has the disadvantage of heating the zinc coating, which, however, in most cases does not prevent the use of the method.

After mounting, the specimens are sectioned by sawing and leveling off with a file. They are then rubbed down first on No. 2 carborundum cloth followed by No. 0, No. 00, and No. 000 Huberts' French emery papers. The wet polishing is done on the usual polishing wheels in three steps: (1) SFX emery powder on a good grade of white duck canvas; (2) 6X emery powder on broadcloth; (3) 10X emery on broadcloth. The emery powders are first shaken up in a bottle with water and applied to the wheels in the form of a thin mud. A moderate wheel speed and plenty of water should be used. It is not advisable to attempt to polish out all the scratches in the steel base, since the shortest possible polishing time is desired in order to reduce the inevitable difference in level between the soft zinc coating and the hard steel base. For the same reason, if it is possible, it is well to avoid having the scratches parallel the line of contact between the steel base and the coating at any time during the polishing operations.

Before etching, the specimens should be thoroughly washed first in ether, then in alcohol, and finally, in water. After shaking or blowing off the excess water from the surface of the specimen, the specimen should be immersed and agitated for about eight seconds in the following etching solution: 100 cc. water, 20 g. c. p. chromic acid (free from sulfates), 1.5 g. sodium sulfate (anhydrous). When the specimen is removed from the etching solution it should be *immediately* washed in a stream of running water, care being taken that the stream impinges first on the polished face of the mount. Enough etching solution should be used in the case of large mounts and specimens mounted in clamps so that the polished face is totally immersed at all times; otherwise disturbing tarnish films may be developed over the surface of the specimens.

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[1] This method of mounting was first demonstrated to the author by Mr. F. M. Crapo, Chief Engineer of the Indiana Steel and Wire Co.

STRUCTURAL CONSIDERATIONS

(HOT-DIPPED GALVANIZED COATINGS)

The constitutional diagram of zinc-iron alloys, as given by Raydt and Tammann,[1] and further amplified by Peirce,[2] can be made to account for the existence of a number of structural fields or constitutents which might be considered to be present in a galvanized coating produced by the hot-dip process. First, there is iron containing zinc in solid solution, second, an iron-zinc constituent having a composition corresponding to $FeZn_3$, third, a phase whose composition varies from 10.9 per cent of iron ($FeZn_7$) to 7.5 per cent of iron and finally there is zinc with not more than 0.01 per cent of iron in solid solution.

It is questionable just how much we are justified in interpreting what the microstructure reveals in terms of the constitutional diagram. In the determination of such diagrams careful consideration must be given to temperature control and the allowance of sufficient time for equilibrium to be reached at every given set of conditions. This practice can hardly be said to be duplicated in the ordinary commercial "hot-dip" galvanizing process. Furthermore, we do not have as yet means of definitely identifying either of the constituents $FeZn_3$ or $FeZn_7$.

The author prefers to consider the various structural features on the basis of only what can be seen when a micro-examination is made. Referring to Fig. 4 it is possible to distinguish the following components of the coating:

1. A layer *A* of relatively pure zinc.
2. A fairly wide band *B* of duplex structure consisting of a hard, zinc rich, iron-zinc constituent more or less dispersed in a matrix of *A*.
3. A narrower layer *C* consisting entirely of a zinc rich iron-zinc constituent, hard, crystallin and light colored.
4. A very thin layer *D* which is also a hard, crystallin, iron-zinc constituent, darker colored and less rich in zinc than *C*.
5. Finally *E* the steel base.

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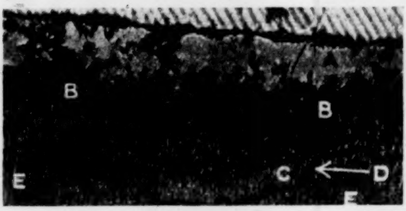
[1] *Loc. cit.*

[2] W. M. Peirce, "Studies on the Constitution of Binary Zinc Base Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 68, p. 772.



A.—Relatively pure zinc.
B.—Duplex structure, hard iron zinc constituent dispersed in matrix of *A*.
C.—Iron-zinc constituent, hard crystallin, light colored.
D.—Iron-zinc constituent, also hard and crystallin. Less rich in zinc and darker colored than *C*.
E.—Steel base.

FIG. 4.—Heavy Zinc Coating on Steel Sheets, Produced by "Hot-Dip" Process, Showing Four Constituents.

FIG. 5.—Typical of heavy coatings (2-oz. per sq. ft. of sheet) on pure iron and low carbon steel.

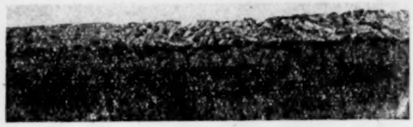


FIG. 6.—Coating on medium carbon steel.

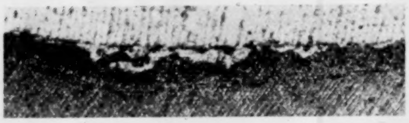


FIG. 7.—Commercial (1.25 oz.) coating on pure iron sheet.

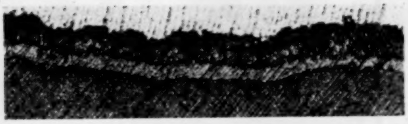


FIG. 8.—Same sheet shown in Fig. 7 after heating for 2 hours at 350° C.

FIG. 9.—Same as Fig. 7 after heating for 4 hours at 350° C.

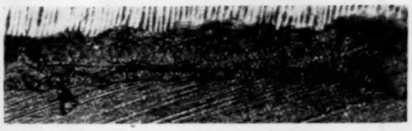


FIG. 10.—Same as Fig. 7 after heating for 5 hours at 350° C.

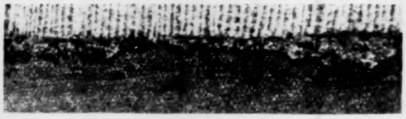


FIG. 11.—Light weight coating (1.2 oz.) on mild steel base, suitable for forming operations.

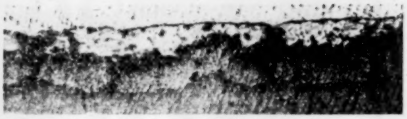


FIG. 12.—Same sheet as Fig. 11 after right angle bend with ⅛ in. radius fillet. Cracking, but no flaking.

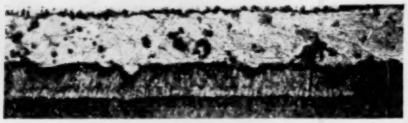


FIG. 13.—Medium heavy coating (1.5 oz.) on mild steel base.

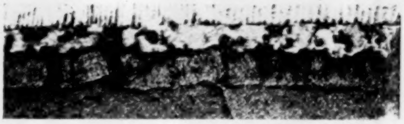


FIG. 14.—Same sheet as Fig. 13, after right angle bend with ⅛ in. radius fillet. Cracking with peeling of coating from base.

FIGS. 4–14.—Photomicrographs of Hot-Dip Galvanized Coatings. (× 350, Reduced One-third in Reproduction.)

It is seldom that all of the foregoing structural features are easily distinguishable in one section as shown in Fig. 4. Microstructures similar to those shown in Figs. 5, 6 and 7 are more often encountered in the examination of ordinary commercial hot galvanized coatings on iron or steel sheets.

The free use of the terms $FeZn_3$ and $FeZn_7$ to designate the alloy constituents of a galvanized coating is open to some criticism if the presence of a definite chemical compound is implied. For example, in structures similar to that shown in Fig. 5 the wide band of alloy is frequently designated as $FeZn_7$ and the very narrow band next to the steel base as $FeZn_3$. While admitting that this may be the correct interpretation of the structure, it is the author's belief, at least in reference to this particular section, that the narrow band is an iron zinc compound probably of the composition $FeZn_7$ while the darker colored wide band of alloy is the duplex structure *B* previously referred to. The columnar-like crystals of iron-zinc constituent in this layer may closely approach the composition $FeZn_7$ at their base and become less rich in iron as they grow out into the matrix of zinc.

The evidence supporting this belief is shown in Fig. 4 and also in the microstructures secured by heating a galvanized coating similar to that shown in Fig. 5. Samples of commercial hot-galvanized sheet were heated for various lengths of time at a temperature of 350° C. in a container filled with zinc dust to cut down surface oxidation of the coating. Under these conditions the alloy layers will grow as iron diffuses into the coatings. Fig. 7 shows the original sheet before heating and Figs. 8, 9 and 10 show the growth of the alloy constituents at various stages during the heating cycle. In Figs. 9 and 10 a narrow band of alloy next to the steel can be seen which was not discernible in the original structure before heating. This is believed to be the compound $FeZn_3$. It may be present in every galvanized coating produced by the hot-dip process but not always developed to a sufficient extent to be easily recognized with our present technique of polishing and etching.

A satisfactory answer of this question probably will not be given until such a time as we have developed a metallographic technique which permits the positive identification of the compounds $FeZn_3$ and $FeZn_7$.

On the basis of convenience there is some justification for the use of the terms $FeZn_3$ and $FeZn_7$ to indicate in general the iron-zinc ratio and it is in this sense that they are used throughout the rest of this paper.

MICROSTRUCTURE VERSUS PHYSICAL PROPERTIES OF ZINC COATING

There is every reason to believe that the physical properties of the coating, so far as its resistance to corrosion and its malleability is concerned, are related to its microstructure. Messrs. Hocker and Farnsworth[1] have already shown that the rate of corrosion of hot-dipped galvanized coatings depends to a great extent upon the presence and amount of certain iron-zinc alloys. Unfortunately, all the relations existing between the microstructure and the bending properties of the zinc coating are not easily explained and in many cases not even known. There are, however, a number of factors, whose significance we are just beginning to appreciate, which have a marked influence on both the type of microstructure produced and the bending properties of the coating.

It is becoming more and more evident that the conditions existing at the surface of the steel after the heat treating, pickling and fluxing operations preparatory to galvanizing are completed have a marked effect on the type of microstructure that results. The chemical composition of the steel is also an important factor, both in respect to its influence on the type of surface produced by pickling operations as well as the probable influence that the carbon, phosphorus, sulfur, etc., in the steel have on the growth of alloy layers. This is illustrated in part by the microstructures shown in Figs. 5 and 6 and also Figs. 19 and 29. Apart from the effects produced by the foregoing variables, the type of microstructure produced is also dependent on the temperature of the zinc bath and the length of time the article remains in it during the galvanizing operation (see Fig. 17(*b*)). The composition of the bath is likewise an important factor. In addition, in the case of wire, the type of structure will depend upon the treatment the wire receives after it leaves the zinc bath, that is, whether it is drawn through charcoal (so-called double galvanized or unwiped wire) or through an asbestos wipe under pressure, or unwiped and heat treated. The space limitations of this paper do not permit a more detailed discussion of all these points. However, the effects of several of the variables, cited in the foregoing paragraph, are illustrated in a number of the photomicrographs.

A few general observations on the relation of microstructure to bending properties can be made which hold true in a large majority of the cases:

1. In general the thicker the zinc coating the more liable it is to peel when subjected to severe deformation (see Figs. 13, 14, 25 and

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[1] An informal paper presented at the general meeting of Committee A-5, March 18, 1926, Providence, R. I.

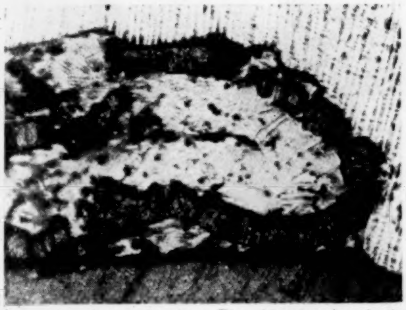

FIG. 15.—Same sheet as Fig. 13, showing bad peeling of the coating after severe forming.

FIG. 16.—Coating high in tin, diagonal streaks in pure zinc part of coating tin-zinc eutectic.

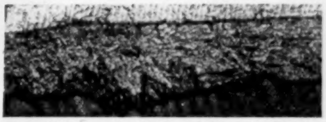

FIG. 17(*a*).—Coating produced with 0.2 per cent aluminum in zinc bath. No visible alloy layers.

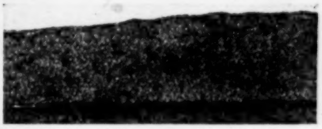

FIG. 17(*b*).—Wire galvanized in $\frac{1}{20}$ sec. Experiment to show time factor on growth of alloy.

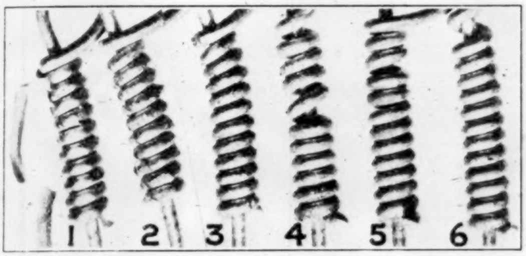

FIG. 18.—No. 12 gage wires wrapped on own diameter to test adherence and malleability of zinc coating; specimens arranged in order poorest No. 1 to best No. 6.

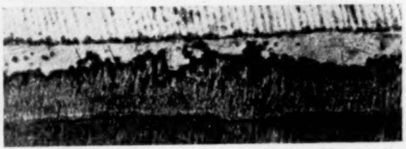

FIG. 19.—E.B.B. zinc-coated wire. No. 1 in Fig. 18.

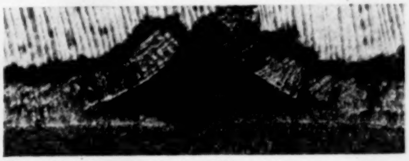

FIG. 20.—Same wire as Fig. 19, wrapped on mandrel 3 times diameter of wire. Bad cracking and peeling.

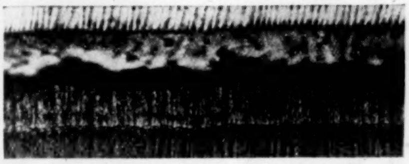

FIG. 21.—Mild steel zinc-coated wire. No. 2 in Fig. 18.

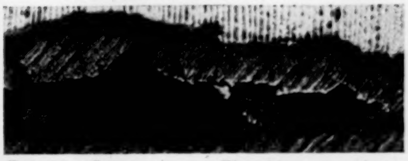

FIG. 22.—Same wire as Fig. 21, wrapped on mandrel 3 times diameter of wire. Coating flaking.

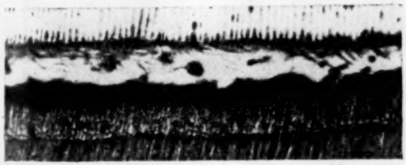

FIG. 23.—B.B. zinc-coated steel wire. No. 3 in Fig. 18.

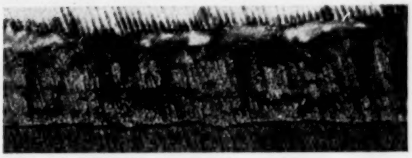

FIG. 24.—Same wire as Fig. 23, wrapped on mandrel 3 times diameter of wire. Coating cracked.

FIGS. 15-24.—Photomicrographs of Hot-Dip Galvanized Coatings. (× 350, Reduced One-third in Reproduction.)

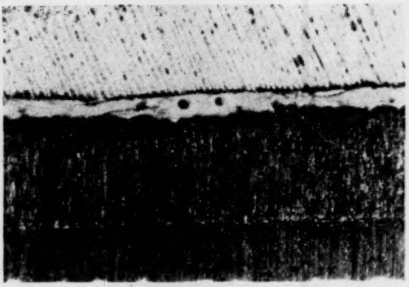

FIG. 25.—Bessemer steel wire, very heavy zinc coating 1.5 oz. per sq. ft. of surface. No. 4 in Fig. 18.

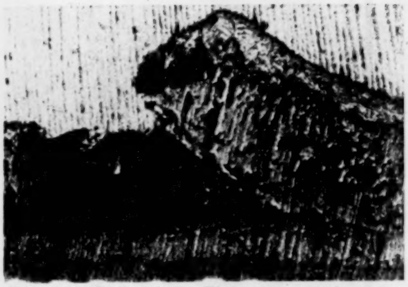

FIG. 26.—Same wire as Fig. 25, wrapped on own diameter. Note separation between alloy layers.

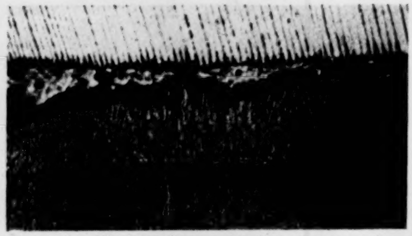

FIG. 27.—Zinc coating on 30–40 per cent carbon steel wire. No. 5 in Fig. 18.

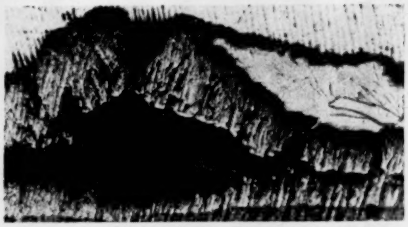

FIG. 28.—Same wire as Fig. 27, wrapped on own diameter. Coating checks, but no flaking. Note some alloy still adhering to steel base.

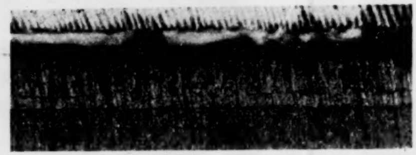

FIG. 29.—Zinc coating on 50–60 carbon steel wire. No. 6 in Fig. 18.

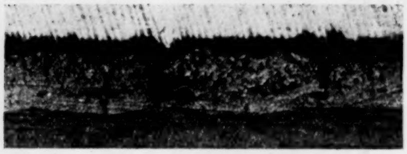

FIG. 30.—Same wire as Fig. 29, wrapped on own diameter. Coating shows fine hair cracks. Note cracks penetrate first alloy layer only.

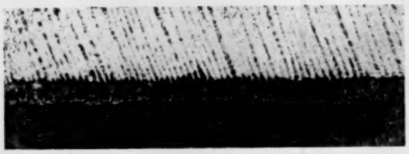

FIG. 31.—"Tight" wiped wire coating (0.26 oz). composed entirely of zinc iron alloy.

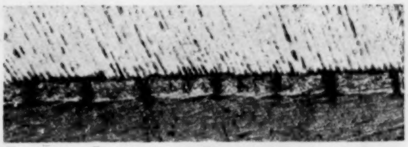

FIG. 32.—Same wire as Fig. 31, wrapped on own diameter. Cracking, but no flaking.

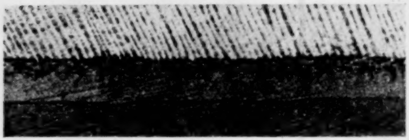

FIG. 33.—Asbestos wiped wire, coating (0.65 oz.) all zinc-iron alloy.

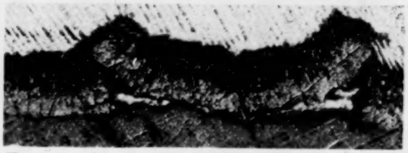

FIG. 34.—Same wire as Fig. 33, wrapped on own diameter. Cracking, some flaking.

FIGS. 25–34.—Photomicrographs of Hot-Dip Galvanized Coatings. (× 350, Reduced One-third in Reproduction.)

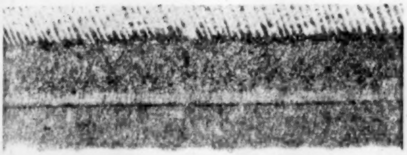

FIG. 35.—Type of coating secured with unwiped hot galvanizing followed by heat treatment. Coating (0.6 oz.) all iron-zinc alloy.

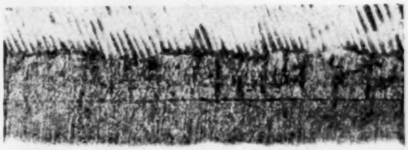

FIG. 36.—Same wire as Fig. 35, wrapped on own diameter. Many fine cracks, but no peeling or flaking.

FIG. 37.—Same type wire as Fig. 35. Coating (0.7 oz.) all iron-zinc alloy.

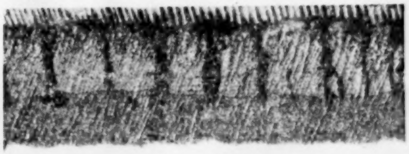

FIG. 38.—Same wire as Fig. 37, wrapped on own diameter. Many fine cracks, no peeling or flaking.

FIGS. 35–38.—Photomicrographs of Hot-Dip Galvanized Coatings. (× 350, Reduced One-third in Reproduction.)

FIG. 39.—Zinc coating produced by the sherardizing process. All zinc-iron alloy 0.9 oz. per sq. ft. of surface. (× 350, reduced one-third in reproduction.)

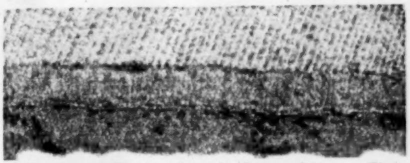

FIG. 40.—Electro-deposited coating. Zinc coating 0.86 oz. per sq. ft. of surface No alloy. (×100, reduced one-third in reproduction.)

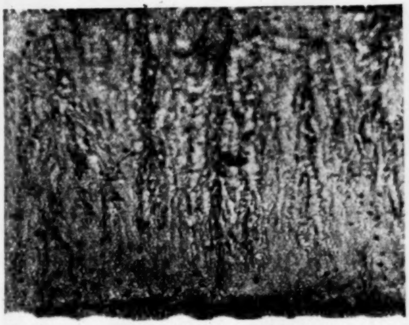

FIG. 41.—Same as Fig. 40. (× 500, reduced one-third in reproduction.)

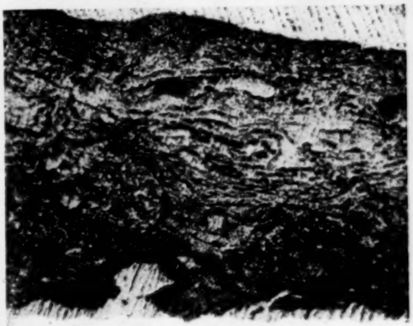

FIG. 42.—Sprayed (Schoop Process) zinc coating, very irregular, average 0.97 oz. per sq. ft. of surface. No visible alloy; note inclusions of oxide.

FIGS. 39–42.—Photomicrographs of Zinc Coatings Produced by the Sherardizing, Electro-Galvanizing and "Schoop" Spray Processes.

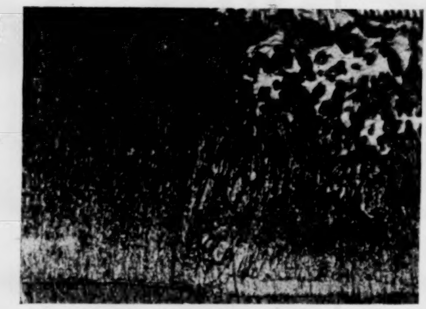

FIG. 43.—Steel, Carbon 0.11 per cent, Phosphorus 0.28 per cent, Sulfur 0.058 per cent, Silicon 0.061 per cent, Manganese 0.55 per cent, Copper 0.040 per cent.

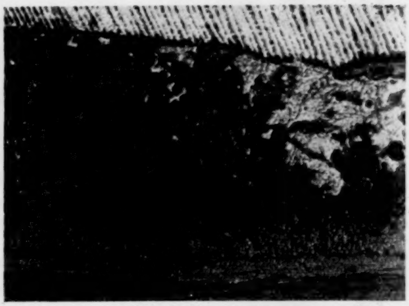

FIG. 44.—Steel, Carbon 0.11 per cent, Phosphorus 0.028 per cent, Sulfur 0.059 per cent, Silicon 0.056 per cent, Manganese 0.55 per cent, Copper 0.040 per cent.

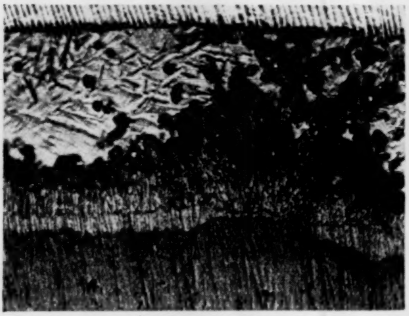

FIG. 45.—Steel, Carbon 0.20 per cent, Phosphorus .008 per cent, Sulfur 0.030 per cent, Silicon 0.019 per cent, Manganese 0.59 per cent, Copper 0.012 per cent.

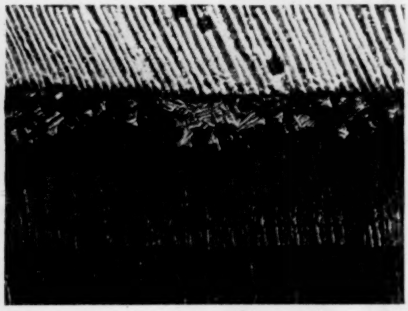

FIG. 46.—Steel, Carbon 0.21 per cent, Phosphorus 0.102 per cent, Sulfur 0.030 per cent, Silicon 0.061 per cent, Manganese 0.68 per cent, Copper 0.032 per cent.

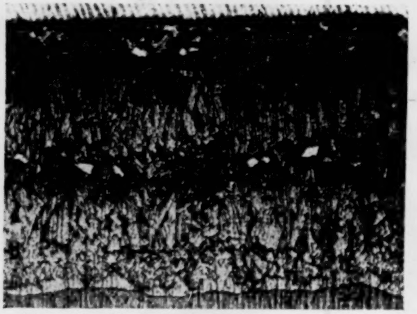

FIG. 47.—Steel, Carbon 0.36 per cent, Phosphorus 0.011 per cent, Sulfur 0.028 per cent, Silicon 0.131 per cent, Manganese 0.52 per cent, Copper 0.012 per cent.

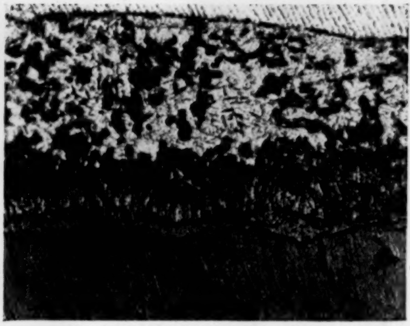

FIG. 48.—Pure iron, Carbon 0.027 per cent, Phosphorus 0.008 per cent, Sulfur 0.049 per cent, Silicon 0.01 per cent, Manganese 0.008 per cent.

FIGS. 43–48.—Photomicrographs of Zinc Coatings Produced by Hand-Dip Galvanizing with Steels of Various Compositions. (× 350, Reduced One-third in Reproduction.) Bars, $\frac{1}{4}$ by 2 by 18 in.

26). This is true even with asbestos wiped wires consisting almost entirely of iron-zinc alloy (see Figs. 33 and 34). Conversely, thin coatings in general have good bending properties (see Figs. 11, 12, 31 and 32). However, by carefully controlling certain conditions of the galvanizing process, heavy coatings can be produced on wires which will withstand severe bending.

2. An irregular alloy layer, similar to that shown in Fig. 5, is usually associated with poor bending properties, and a smooth even alloy layer (see Fig. 7) with good bending properties. An irregularity in the line of contact between the steel and the alloy layers is also suspected of producing poor bending properties.

3. The presence of cadmium and lead in the zinc bath influences, to a marked extent, the ductility of the pure zinc part of the coating. This point is of great importance in securing ductile coatings on the heavily coated (one ounce per square foot of surface) wires galvanized by the "charcoal wiped" process. So far as the author knows, ductile coatings of this sort can only be obtained with high grade zinc having a cadmium content of less than 0.03 per cent. Lead in excess of 0.1 per cent is also considered by some galvanizers to be detrimental to the ductility of such coatings.

Figs. 39 to 42, inclusive, illustrate the microstructure of zinc coatings produced by the sherardizing, electrogalvanizing and zinc spray (Schoop) processes. The malleability of these coatings is of little importance since they are generally applied to the surface of the iron or steel object after all fabricating operations are completed. Their structure, however, may be worth studying if one is investigating the corrosion resistance of these types of zinc coatings.

The microstructures illustrated by Figs. 43 to 48, inclusive, are included to illustrate the great thickness of zinc coatings produced by the "hand dip" process. They also show the wide variety of forms taken by the zinc-iron alloy constituents which are probably due, in part at least, to the differences in composition of the steel base.

Acknowledgement.—The author is especially indebted to Mr. W. M. Peirce, Mr. A. C. Simmons and other members of the Metal Section staff, Research Division, New Jersey Zinc Co., whose kind assistance has made possible the preparation of this paper. He is also indebted to Mr. F. M. Crapo, Chief Engineer, Indiana Steel and Wire Co., who generously supplied a number of the galvanized wire samples.

DISCUSSION

Mr. Kemp.

MR. J. T. KEMP.[1]—I should like to ask Mr. Finkeldey whether the light band which appears on the steel base in a good many of the slides he has shown us, is an etching effect or a structure, and whether if it is an etching effect, it is due to any porosity in the iron-zinc compound alloy just above the steel base which might hold the etching solution after the samples were etched and washed.

Mr. Finkeldey.

MR. W. H. FINKELDEY.—Where a zinc coating has been stressed as in the case of bent specimens there is a possibility that the alloy layers may separate from the steel base. In etching a micro-section from such a specimen, some solution might be retained in this crack and produce a staining effect on the steel which might be mistaken for an iron-zinc alloy layer. However, I believe in most cases the light band referred to by Mr. Kemp is one of the iron-zinc compounds. Unfortunately, due to the difference in elevation between the steel, iron-zinc alloy layer and pure zinc part of the coating, in many instances it is impossible, with our present polishing and etching technique, to prepare sections so that this narrow alloy band can be clearly seen and not mistaken for an etching effect or a shadow cast by the projecting steel base.

Figures 4 and 7 show structures having this narrow band of alloy. That this is actually a zinc alloy, and not a shadow or etching effect, is proved by the type of structure secured after heating a galvanized sheet in order to produce a growth of the alloy layers. This experiment was carried out with the sheet whose structure is illustrated in Fig. 7. The increase in thickness of the narrow alloy band can be easily traced at various stages during the heating by referring to Figs. 8, 9 and 10. The original narrow band of alloy has grown until it is approximately equal to the thickness of the entire coating and a second narrow band of iron-zinc alloy produced, which was not distinguishable in the structure of the original sheet.

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[1] Technical Department, The American Brass Co., Waterbury, Conn.

THE ETCHING CHARACTERISTICS OF CONSTITUENTS IN COMMERCIAL ALUMINUM ALLOYS

By E. H. Dix, Jr.,[1] and W. D. Keith.[2]

Synopsis

The industrial importance of aluminum alloys and the present scientific interest in the study of their microscopic structure suggests the need of a systematic presentation of the etching characteristics of the constituents composing these structures. A survey of the recent literature will show that little information is available on this important subject and also that there has been little effort to supplement or organize the data. The experience of the laboratories of the Aluminum Company of America in studying the structures of many special and commercial alloys has been supplemented by a systematic study of the etching characteristics of some twenty-eight specially prepared alloys and the information obtained is summarized in the present paper.

General Occurrence of Constituents

The commercial light alloys generally contain 85 per cent or more of aluminum, the balance being made up of elements which are added to strengthen or harden the metal, or else to impart to it some specific property. In addition, iron, silicon, and a small amount of copper are always present as impurities. These elements all form compounds with aluminum, silicon being the only important exception. Such compounds and silicon form eutectics with the aluminum and thus appear as the so-called constituents in the microstructure.

Figs. 1 to 4 will illustrate the general type of structure found in the cast alloys. The particular structure shown is that of an aluminum alloy containing 4 per cent of copper, made from metals of high purity, chill cast in a cold graphite mold. For revealing the macro or grain structures shown in the first two figures, the specimen has been etched with a hydrofluoric-hydrochloric acid mixture described in a previous publication from this laboratory.[3]

The entire cross-section of the $\frac{3}{4}$-in. diameter bar is included in Fig. 1. The oblique illumination reveals the individual grains in varied tones. Each grain consists of a dendrite of aluminum-rich solid solution, several orders of axes giving rise to a dendritic structure as illustrated in Fig. 2. The interstices between the branches of the dendrite are filled with the eutectic mixtures. The dark, irregular grain to the left center of Fig. 2 may be located in the lower left of Fig. 1 by means of the prominent projection resembling an animal's nose. Because of the different illumination this grain appears light in Fig. 1 and dark in Fig. 2. The eutectic network partially surrounding the aluminum-rich areas is illustrated in Fig. 3, in which the "nose" is in the upper half of the micrograph; whereas Fig. 4 is at sufficient magnification to show the particles of the compound $CuAl_2$ which have formed a eutectic with the surrounding aluminum solid solution. The large, irregular cluster in the lower righthand corner of the figure may be located just below the "nose" in Fig. 3. Since this is an "as cast" and unannealed structure, the cored nature of the aluminum solid solution is evident in the last two micrographs.

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[1] Metallurgist, Research Bureau, Aluminum Co. of America, New Kensington, Pa.

[2] Assistant Metallurgist, Research Bureau, Aluminum Co. of America, New Kensington, Pa.

[3] F. B. Flick, "Etching Aluminum Alloys for Macroscopic and Microscopic Examination," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. LXXI, pp. 816-827 (1925).

In less pure and more complicated alloys, particles of the constituents formed by the impurities and various added elements will be found in the same network structure. For obvious reasons it is desirable to be able to identify the particles of constituents as they occur either in the eutectic network in cast structures, or in fabricated alloys as uniformly oriented particles formed by the breaking up of the original eutectic network.

GENERAL CHARACTERISTICS OF CONSTITUENTS

Many of the constituents commonly occurring in the commercial alloys are readily distinguished by their color and polishing characteristics, but since they occur in alloys containing other constituents less easily identified and, therefore, requiring etching, it is necessary to know the effect of the etching reagents on the easily identified constituents, because their appearance is often changed in a very marked manner by the etching reagent. In the first group of the constituents would be classed silicon, $CuAl_2$, Mg_2Si, and Mg_2Al_3. In the second group of constituents are included a number of light gray constituents which may occasionally be differentiated in the unetched specimen when two or more different particles occur in the same field of the microscope, but this distinction is impossible when they are more widely separated or occur in different specimens. The important constituents in this group are $MnAl_3$, $NiAl_3$, and an uncertain group of constituents, due at least in part, to the presence of iron as an impurity in all commercial alloys. It has been customary in our laboratories to group the latter under the general heading of iron constituents.

THE COMPLEX CONSTITUENTS

The present experiments have thrown considerable light on the nature of these constituents. It is, of course, well known that in the high purity binary aluminum-iron alloys, iron occurs as the compound $FeAl_3$.[1] The effect of the addition of small amounts of silicon in changing the characteristic form of this constituent has been discussed previously.[2] The experience of the authors with constituents due to the presence of both iron and silicon seems to indicate that the composition of the constituent so formed varies through wide limits, and a very thorough study of the constitution of the ternary aluminum-iron-silicon system will be required before definite limits can be placed on its composition. It has also been found that the nature of the iron constituent, judging from the etching characteristics, is altered in a similar manner by the presence of manganese, copper, and perhaps other elements. The authors, therefore, propose to treat each of these as an individual constituent tentatively and have named them according to the fewest elements required to produce them. Thus, the iron constituent, believed to contain some manganese, has been designated as the Fe-Mn constituent and in this manner the so-called "X" constituent, now generally conceded to contain iron, silicon and aluminum, as Fe-Si constituent. Several other constituents are designated in a similar manner, namely, the so-called "T" constituent,[3] containing copper, nickel and aluminum, which we designate as Cu-Ni; and a constituent due to the simultaneous presence of manganese and silicon, designated as Mn-Si; and lastly, a constituent of rather complex composition which has been found in alloys of copper, manganese, iron, and aluminum as the Cu-Fe-Mn constituent.

SELECTION OF ALLOYS

In order to study the etching characteristics of the constituents as outlined above and to determine the effect, if any, of the presence of one constituent on the etching characteristics of the other, a series of some 28 alloys was prepared. By the use of aluminum of high purity, it has been possible to prepare alloys containing only one added element at a time, the total impurities being less than 0.10 per cent and in most cases less than 0.05 per cent. The constituents thus obtained were $FeAl_3$, silicon, $CuAl_2$, $MnAl_3$, Mg_2Al_3, and $NiAl_3$. By the addition of magnesium and silicon in the proper ratio the compound Mg_2Si was obtained. The nature of the various iron constituents was investigated by adding manganese, silicon, and copper, respectively, to an aluminum-iron mixture. Manganese and silicon were added together, with and without iron. In this way various combinations of elements were obtained, starting with a simple binary alloy and leading up to one of the most complicated commercial alloys, namely, that of the duralumin type containing copper, iron, silicon, manganese, and magnesium. The constituents formed by other elements such as nickel, zinc, tin, chromium, vanadium, etc., excepting the binary compound $NiAl_3$ and the "T" constituent, have not been included within the scope of this investigation, because they do not form constituents which are present in the microstructure of the commercially important alloys. The complete list of these alloys with their chemical analyses, fabrication history, and the constituents observed, is given in Table I.

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[1] E. H. Dix, Jr., "A Note on the Microstructure of Aluminum-Iron Alloys of High Purity," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 120 (1925).

[2] E. H. Dix, Jr., "Observations on the Occurrence of Iron and Silicon in Aluminum," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. LXIX, pp. 957–970 (1923).

[3] K. E. Bingham and J. L. Haughton, "The Constitution of Some Alloys of Aluminum with Copper and Nickel," *Journal*, Inst. Metals, Vol. XXIX, No. 1, pp. 71–112 (1923).

TABLE I.—ALLOYS USED IN STUDY OF ETCHING CHARACTERISTICS.

Alloy	Composition, per cent						History of Treatment	Constituents Present
	Cu	Fe	Si	Mn	Mg	Ni		
No. 1	4.10	0.04	0.02	.....	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$
No. 2	4.05	.....	.....	.....	.....	.....	Chill cast in graphite mold	$CuAl_2$
No. 3	0.02	7.07	0.02	.....	.....	.....	Slowly cooled ingot	$FeAl_3$
No. 4	0.10	0.39	12.89	.....	.....	.....	Chill cast	Si, Fe-Si
No. 5	0.01	0.05	0.04	2.96	.....	.....	Chill cast in graphite mold	$MnAl_3$
No. 6	.....	.....	.....	.....	10.21	.....	Chill cast in graphite mold	Mg_2Al_3
No. 7	.....	.....	.....	.....	.....	1.72	Slowly cooled ingot	$NiAl_3$
No. 8	3.89	0.50	0.02	.....	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, $FeAl_3$, Fe-Cu
No. 9	4.05	0.03	0.37	0.00	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, Si
No. 10	4.06	0.17	0.04	.....	.....	2.01	Slowly cooled ingot	$CuAl_2$, $NiAl_3$, Cu-Ni
No. 11	0.22	0.74	0.34	.....	.....	.....	Chill cast notch bar	Fe-Si, $FeAl_3$
No. 12	.....	1.02	0.42	.....	.....	.....	Chill cast in iron mold—annealed[2]	Fe-Si, $FeAl_3$
No. 13	0.28	13.25	14.43	0.01	.....	.....	Chill cast in graphite mold	Fe-Si, $FeAl_3$, Si
No. 14	0.03	0.63	0.02	0.77	.....	.....	Slowly cooled ingot—annealed[1]	Fe-Mn, $MnAl_3$, $FeAl_3$
No. 15	.....	0.68	0.03	0.00	0.83	.....	Slowly cooled ingot—annealed[1]	$FeAl_3$
No. 16	0.01	0.04	0.51	2.10	.....	.....	Slowly cooled ingot	$MnAl_3$, Mn-Si, Si
No. 17	0.01	0.07	1.82	2.12	.....	.....	Chill cast in graphite mold	$MnAl_3$, Mn-Si, Si
No. 18	.....	.....	.....	0.75	0.83	.....	Slowly cooled ingot—annealed[1]	$MnAl_3$.
No. 19	0.02	0.10	11.88	0.01	20.15	.....	Chill cast in graphite mold	Mg_2Si, Si
No. 20	3.83	0.50	0.44	0.00	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, Fe-Cu, Si ($FeAl_3$ or Fe-Si)
No. 21	3.72	0.62	0.02	0.77	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, Cu-Fe-Mn, Fe-Mn
No. 22	0.03	0.62	0.61	0.75	.....	.....	Slowly cooled ingot	Fe-Mn, Si, Fe-Si
No. 23	.....	0.68	0.02	0.74	0.87	.....	Slowly cooled ingot—annealed[1]	Fe-Mn, $MnAl_3$, $FeAl_3$
No. 24	3.92	0.49	0.43	0.59	.....	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, Si, Cu-Fe-Mn
No. 25	.....	0.64	0.59	0.65	0.89	.....	Slowly cooled ingot	Mg_2Si, Si, Fe-Mn, $MnAl_3$
No. 26	.....	0.64	1.61	0.62	0.84	.....	Slowly cooled ingot	Mg_2Si, Si, Fe-Mn, $MnAl_3$ (Fe-Si or $FeAl_3$)
No. 27	3.52	0.48	0.42	0.55	0.46	.....	Slowly cooled ingot—annealed[1]	$CuAl_2$, Si, Mg_2Si, Cu-Fe-Mn
No. 28	4.04	0.50	0.55	0.59	0.48	.....	14 gage sheet—quenched from 510° C.	$CuAl_2$, Cu-Fe-Mn

[1] Annealed at 455° C. for 12 days and slowly cooled to room temperature through a period of 5 days.
[2] Annealed 175 hours at 550° C. and quenched.

SELECTION OF ETCHING REAGENTS

The etching reagents which experience has shown to be of importance in studying the microstructure of aluminum alloys are comparatively few. Reference should be made to the bibliography on etching reagents for aluminum alloys, together with their recommended use and authority, which has been prepared as a tentative revision of the A.S.T.M. Standard Methods of Metallographic Testing of Non-Ferrous Metals and Alloys (Serial Designation, E 5 – 24).[1] Special recommendations for preparation of aluminum specimens are also included in these revisions. Two of these etchings reagents, namely, hydrofluoric acid and sodium hydroxide, have been used for general purposes and a few special reagents have been recommended for specific purposes. In the early stages of the development of aluminum metallography, it was generally necessary to use some etching reagent in order to reveal the presence of undissolved constituents. For this purpose various concentrations of hydrofluoric acid and sodium hydroxide were used and in the literature may be found many conflicting statements as to the effect of these reagents on the various constituents. As the polishing technique was improved, it became less imperative to use an etching reagent in order to reveal the microstructure. For instance, several methods have been recommended for differentiation between $CuAl_2$ and the iron constituents. However, with proper polishing technique, these are readily differentiated without etching because of the difference in color. Thus, etching for this purpose is unnecessary, although the contrast may be improved for photographic purposes by a light swabbing with a dilute solution of hydrofluoric acid. Generally, however, photographs of the unetched structures present a much more pleasing appearance. Compare, for instance, Fig. 4 and Fig. 10. Both of these specimens were prepared with equal care and the structure of Fig. 4 had the same smooth surface before etching. The staining of the cored aluminum-copper solid solution by the hydrofluoric acid etch is particularly troublesome. This stain may be removed by a subsequent treatment with concentrated nitric acid, but this removes the evidence of the cored structure and in addition spoils the distinguishing color produced on some of the other constituents by the hydrofluoric acid etch.

In this investigation, the authors are primarily concerned with the differentiation between constituents which in the unetched condition are not readily identified. This, of course, concerns the light gray constituents, and etches which are proposed for attacking the other constituents are not recommended. Hydrofluoric acid and sodium hydroxide both readily dissolve the aluminum solid solution matrix and the more concentrated solutions rapidly attack most of the constituents, which are thus eaten out and all possibility of identification lost. However, dilute solutions of both reagents may be selected which will dissolve sufficient aluminum to remove all surface flow while the attack on the constituents will be so light as to afford a satisfactory means of identification in several instances.

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[1] See Report of Committee E-4 on Metallography, p. 565 *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 565 (1926).

Before dealing with the specific reagents recommended, a few general precautions concerning etching practice should be emphasized. Two general methods of etching have been recommended, that is, immersion and swabbing. Previously the common method of etching aluminum alloys has been that of immersion. Within the last few years this laboratory has made a practice of swabbing with a soft cotton swab when using dilute solutions of hydrofluoric acid or sodium hydroxide. This method has been found to give uniform and satisfactory results and is especially desirable in preparing a surface for photography, since there is less danger of pitting or uneven staining. The method of swabbing is quite commonly used for other alloys, but has not been generally used for aluminum alloys. The importance of the effect of temperature, both of the etching solution and specimen, has not been fully realized. These experiments have shown that it is very important to control the temperature, both of specimens and solution, the concentration, and the time of etching. Most of the literature dealing with etching of aluminum alloys is lamentably lacking in respect to these details.

The following five specific etching reagents are recommended by the authors. These are all aqueous solutions which in the case of the acids are made up by volume, using the commercial concentrated acid, and in the case of sodium hydroxide, by weight.

1. 0.5-per-cent hydrofluoric acid, applied by swabbing for 15 seconds at room temperature.
2. 1-per-cent sodium hydroxide, applied by swabbing for 10 seconds at room temperature.
3. 10-per-cent sodium hydroxide at 70° C. Specimen is immersed for 5 seconds and subsequently rinsed in cold water.
4. 20-per-cent sulfuric acid at 70° C. Specimen is immersed for 30 seconds and subsequently quenched in cold water.
5. 25-per-cent nitric acid at 70° C. Specimen immersed for 40 seconds and subsequently quenched in cold water.

The most generally useful etch is that of swabbing with 0.5 per cent HF. This satisfactorily removes surface flow, revealing any minute particles of constituent and at the same time offers certain possibilities of identification of constituents.

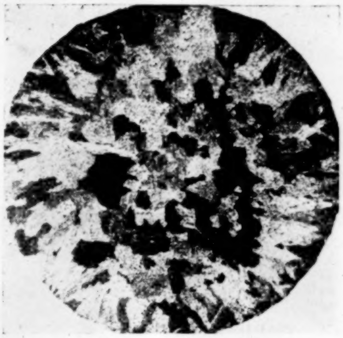

FIG. 1.—ALUMINUM-COPPER ALLOY (NO. 2, Cu 4.05 PER CENT) CHILL CAST IN COLD GRAPHITE MOLD. ENTIRE CROSS-SECTION. ETCHED HF:HCl:H_2O::10:15:90. OBLIQUE ILLUMINATION. (×5, REDUCED ONE-HALF IN REPRODUCTION.)

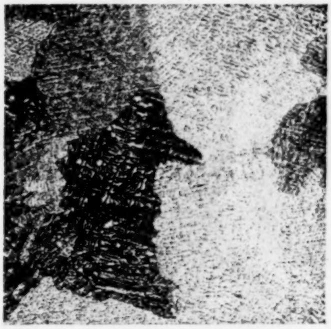

FIG. 2.—SAME SECTION, SHOWING DENDRITIC STRUCTURE OF LOWER LEFT OF FIG. 1. ETCHED HF:HCl:H_2O::10:15:90. (×25, REDUCED ONE-HALF IN REPRODUCTION.)

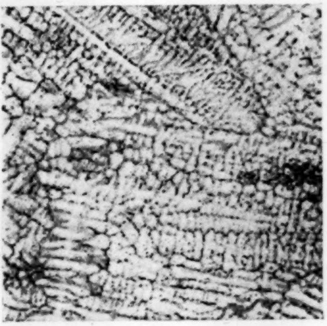

FIG. 3.—SAME SECTION, SHOWING EUTECTIC NETWORK IN LEFT CENTER OF FIG. 2. ETCHED 0.5-PER-CENT HF. (×100, REDUCED ONE-HALF IN REPRODUCTION.)

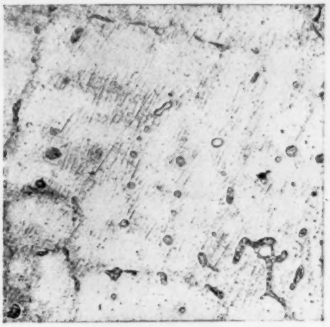

FIG. 4.—SAME SECTION, SHOWING $CuAl_2$ IN LACEWORK AND SOLID PARTICLES AND CORED MATRIX IN CENTER OF FIG. 3. ETCHED 0.5 PER CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

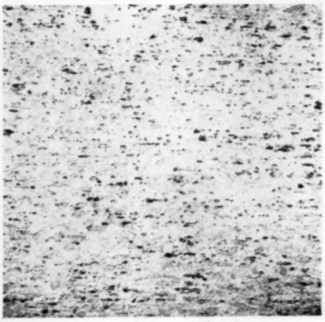

FIG. 5.—HEAT TREATED STRONG ALLOY 14 GAGE SHEET, SHOWING GRAIN SIZE AND ARRANGEMENT OF CONSTITUENT (ALLOY NO. 28). ETCHED 0.5-PER-CENT HF. (×100, REDUCED ONE-HALF IN REPRODUCTION.)

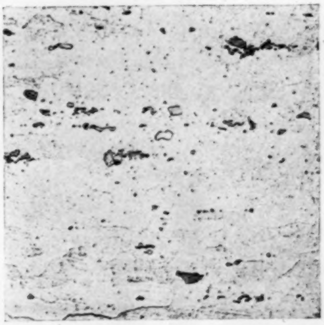

FIG. 6.—SAME SECTION, SHOWING $CuAl_2$, LIGHT WITH DARK BOUNDARY AND Cu-Fe-Mn CONSTITUENT, DARKER. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

Figures 5 and 6 illustrate the very satisfactory manner in which the 0.5-per-cent HF etch can be used to develop the grain structure, at the same time clearly outlining the particles of constituent in a complicated alloy of the duralumin type after heat treatment. In the center of Fig. 6 will be observed an irregular particle, clear with a heavy black boundary, which is $CuAl_2$. The other particles, which appear light gray in the unetched alloy, have been slightly colored by the etch. The 1-per-cent NaOH etch is also satisfactory for removing surface flow, but is not as versatile an aid in identifying constituents.

The last three etches are useful for specific identification.

ETCHING CHARACTERISTICS OF CONSTITUENTS

The effect of these five reagents on the constituents previously described are summarized in Table II. In this table the most satisfactory etching reagent for each constituent, for both identification and photography, has been indicated as explained in the footnote.

The etching characteristics as given in this table are based primarily on the effect of the reagent on the aluminum alloy of high purity containing this constituent alone. However, each of these constituents occurred a number of times in the more complicated alloys and the etching characteristics were the same. It must be emphasized at this point that it is not possible to be absolutely sure that a different etching characteristic, caused by the addition of a third element to a binary aluminum alloy, proves a change in the constituent, since it is conceivable that the action of a particular etch on one constituent may be altered by the electrolytic effect due to another constituent in the microstructure. For instance, neither $FeAl_3$ nor $MnAl_3$ are appreciably affected by the 15-second etch with 0.5-per-cent HF, whereas, if iron and manganese are present together, a gray constituent is found which is colored an even brown by the same etch.

In alloy No. 13, containing 0.63 per cent of iron and 0.77 per cent of manganese, two constituents were observed, one of which etched as the Fe-Mn constituent and the other behaved as $MnAl_3$ did in the pure binary alloy. In other cases the three constituents, $FeAl_3$, Fe-Mn and $MnAl_3$ were found in the same alloy and exhibited normal etching characteristics. Therefore, the authors have concluded that in this case the two elements are contained in the constituent etching brown with 0.5-per-cent HF 15-second swab. However, in an alloy of copper, iron and silicon, a gray constituent is found which behaves in an erratic manner when etched for 15 seconds with 0.5-per-cent HF. At times it is entirely unattacked and at other times colors an uneven

TABLE II.—ETCHING CHARACTERISTIC OF CONSTITUENTS.

Constituent	Reagents				
	0.5-per-cent HF, swab for 15 seconds, wash in cold water.	1-per-cent NaOH, swab 10 seconds, wash in running water.	20-per-cent H_2SO_4 at 70° C., immerse specimen for 30 seconds, quench in cold water.	25-per-cent HNO_3 at 70° C., immerse specimen for 40 seconds, quench in cold water.	10-per-cent NaOH at 70° C., immerse specimen for 5 seconds, rinse in cold water.
Silicon.	Outlined. Unattacked. Color lightened.[a] [b]	Outlined. Unattacked. Colorslightlylightened.	Unattacked. Color lightened.	Outlined. Unattacked. Color lightened.	Outlined. Unattacked. Color lightened.
Mg_2Si.	Colored bright blue (does not always color if particles are tarnished in polishing).[a] [b]	Outlined. Color unchanged.	Action violent. Particles often eaten away. Any particles left have a blue color.[1]	Colored brown or black.	Outlined. Color lightened.
$CuAl_2$.	Outlined. Part of pinkish tinge removed. Constituent light and clear.[a] [b]	Outlined. Part of pinkish tinge removed. Constituent light and clear.	Outlined. Part of pinkish tinge removed. Constituent light and clear.	Colored brown or black.[2]	Pitted. Colored light to dark brown.
Mg_2Al_3.	Outlined. Slightly clearer and more watery. Black pits appear in particles.	Not outlined. to any extent. Unattacked. Uncolored.	Attacked vigorously, resulting in pitting which causes particles to have a dark, eaten out appearance.	Heavy attack, particles grayish and watery.	Outlined. Unattacked. Uncolored.[a] [b]
$FeAl_3$.	Usually uncolored. Brown stains often appear on large primary particles.[b]	Uncolored. Purplish color is slightly faded.	Heavily attacked. Particles often eaten out or deeply pitted. Color darkened.[3]	Outlined. Contrast with Fe-Si improved.[a] [b]	Outlined. Colored deep brown.
Fe-Si Constituent.	Outlined. Not colored.[b]	Outlined. Not colored.	Outlined. Colored black.	Outlined. Contrast with $FeAl_3$ improved.[a] [b]	Attacked. Blackened.
$MnAl_3$.	Outlined. Uncolored.[a] [b]	Attacked. Colored brownish or bluish, but coloring is uneven.	Outlined. Not attacked. Not colored.	Not outlined. Not attacked. Not colored.	Colored blue to brown.
$NiAl_3$.	Outlined. Colored blue and brown.[a] [b] [4]	Outlined. Darkened slightly. Not colored.	Outlined. Darkened slightly. Not colored.	Outlined. Not attacked. Not colored.	Colored blue to deep brown.
Fe-Mn Constituent.	Outlined. Colored brown (sometimes bluish).[a] [b]	Outlined. Particles pitted. (Often a rough blue color on a few particles.)	Outlined. Not attacked. Not colored.[a]	Outlined. Not attacked. Not colored.	Colored deep brown to blue.
Cu-Ni Constituent.	Outlined. Unattacked.[a] [b]	Outlined. Not attacked. Not colored.	Outlined. Not attacked. Not colored.	Eaten out. Blackened.	Outlined. Unattacked. Uncolored.
Fe-Cu Constituent.	Outlined. Not attacked. Not colored. (Resembles $FeAl_3$.)	Outlined. Not colored. Slightly darkened.	Outlined. Uncolored. Often shows black cores, which are probably $FeAl_3$.[a] [b]	Outlined. Uncolored. Unattacked.	Pitted. Colored light brown.
Cu-Fe-Mn Constituent in duralumin types.	Outlined. Colored a light brown to black. Usually appears rough.[a] [b]	Outlined. Uncolored.	Outlined. Colored black.	Outlined. Unattacked. Uncolored.	Outlined. Uncolored.
Mn-Si Constituent.	Outlined. Appears rough and attacked. Darkened in color.	Outlined. Appears rough and attacked. Color unchanged.	Outlined. Appears rough and attacked. Darkened considerably.[a] [b]	Outlined. Appears rough. Darkened.[a]	Outlined. Color not changed.

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[1] For this constituent it may be preferable to use 10 seconds instead of 30 seconds.
[2] For this constituent it may be preferable to use 10 seconds instead of 40 seconds.
[3] For this constituent it may be preferable to use 20 seconds instead of 30 seconds.
[4] Large primary particles in high nickel alloys usually blue.
[a] Recommended etch for identification.
[b] Recommended etch for photographing.

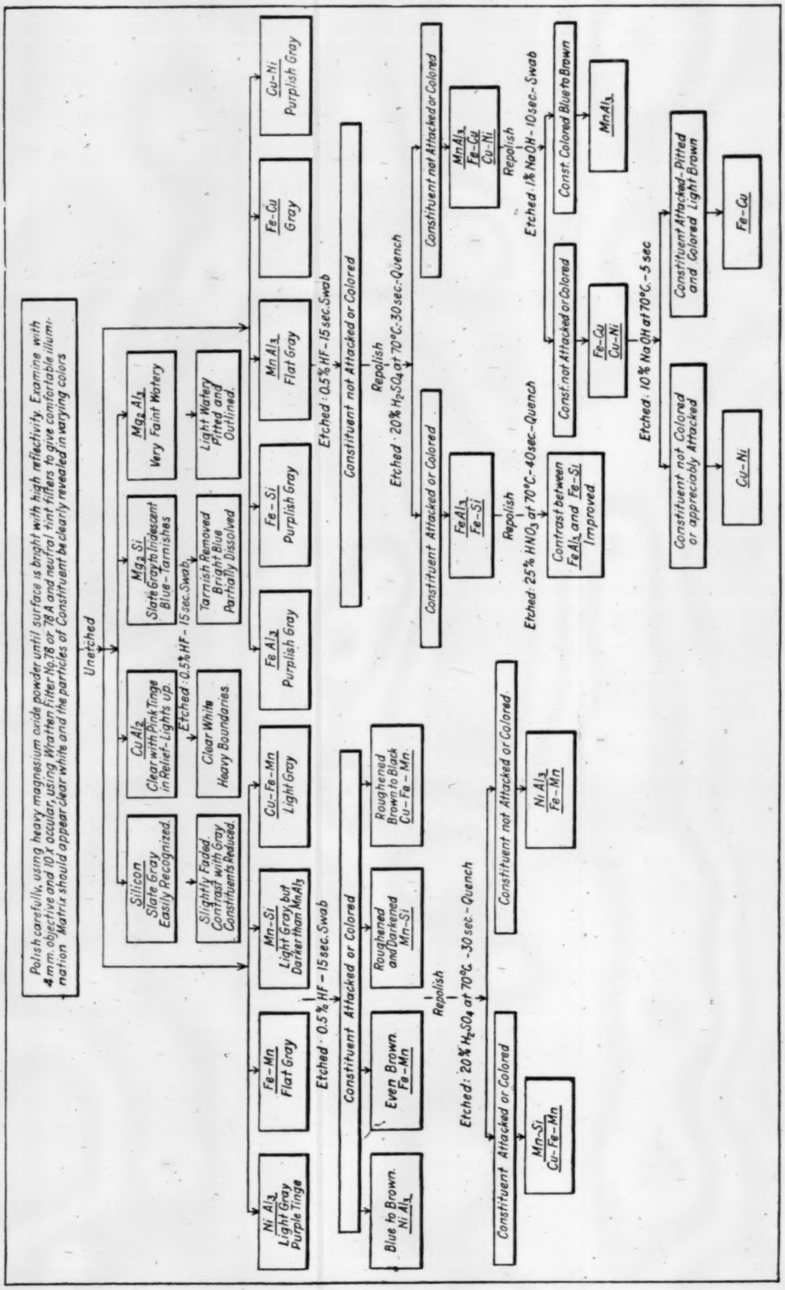
Polish carefully, using heavy magnesium oxide powder until surface is bright with high reflectivity. Examine with 4 mm. objective and 10X occular, using Wratten Filter No. 78 or 78A and neutral tint filters to give comfortable illumination. Matrix should appear clear white and the particles of Constituent be clearly revealed in varying colors
Unetched
Silicon
Slate Gray
Easily Recognized
CuAl2
Clear with Pink Tinge
in Relief - Lights up.
Mg2Si
Slate Gray to Iridescent
Blue - Tarnishes
Mg2Al3
Very Faint Watery
Etched: 0.5% HF - 15 sec. Swab
Slightly Faded.
Contrast with Gray
Constituents Reduced.
Clear White
Heavy Boundaries.
Tarnish Removed
Bright Blue
Partially Dissolved
Light Watery
Pitted and
Outlined.
NiAl3
Light Gray
Purple Tinge
Fe-Mn
Flat Gray
Mn-Si
Light Gray, but
Darker than MnAl3
Cu-Fe-Mn
Light Gray
FeAl3
Purplish Gray
Fe-Si
Purplish Gray
MnAl3
Flat Gray
Fe-Cu
Gray
Cu-Ni
Purplish Gray
Etched: 0.5% HF - 15 sec. Swab
Constituent Attacked or Colored
Blue to Brown.
NiAl3
Even Brown
Fe-Mn
Roughened
and Darkened
Mn-Si
Roughened
Brown to Black
Cu-Fe-Mn.
Repolish
Etched: 20% H2SO4 at 70°C. - 30 sec. - Quench
Constituent Attacked or Colored
Mn-Si
Cu-Fe-Mn
Constituent not Attacked or Colored
NiAl3
Fe-Mn
Etched: 0.5% HF - 15 sec. Swab
Constituent not Attacked or Colored
Repolish
Etched: 20% H2SO4 at 70°C. 30 sec. - Quench
Constituent Attacked or Colored
FeAl3
Fe-Si
Repolish
Etched: 25% HNO3 at 70°C. - 40 sec. - Quench
Contrast between
FeAl3 and Fe-Si
Improved
Constituent not Attacked or Colored
MnAl3
Fe-Cu
Cu-Ni
Repolish
Etched: 1% NaOH - 10 sec. - Swab
Const. not Attacked or Colored
Fe-Cu
Cu-Ni
Etched: 10% NaOH at 70°C. - 5 sec
Constituent not Colored
or appreciably Attacked
Cu-Ni
Constituent Attacked - Pitted
and Colored Light Brown
Fe-Cu
Const. Colored Blue to Brown
MnAl3

FIG. 7.—Guide for Identification of Constituents in Commercial Aluminum Alloys.

brown, and in this case it is suspected that the coloration may be due to the presence of $CuAl_2$ and silicon in the same structure. This effect is well illustrated in the case of etching with 20-per-cent sulfuric acid at 70° C., where it has been found that if the chromel tongs used for handling the specimen are allowed to remain in the solution, the effectiveness of the attack is greatly reduced.

Since this conception of these constituents indicates a solid solution complex, it is to be expected that the etching characteristics may vary somewhat with the composition of the constituent. It should, therefore, be borne in mind that the etching characteristics as outlined in this paper are based upon the constituents occurring in the alloys listed in Table I, and it is quite possible that alloys of somewhat different composition would behave differently. Likewise, most of the alloys examined in practice are in a condition of unstable equilibrium and the etching characteristics of the constituents would very likely be changed by thorough annealing.

SCHEME FOR IDENTIFICATION

A scheme of identification has been prepared to summarize the results obtained in this investigation and to act as a tentative guide for the identification of constituents. It is subject to all of the limitations of any etching procedure and is further complicated by the fact that the constitution of many of the constituents mentioned is uncertain. It is, however, proposed as a working scheme upon which it is hoped may gradually be built a more exact and comprehensive method. In general, the chemical analysis of the specimen under examination is known, at least approximately, and the composition may be used to greatly simplify the scheme of Fig. 7. However, it is believed that very satisfactory conclusions may be obtained by the use of this table, even when the composition of the alloy is totally unknown. It must not be expected that the novice in aluminum metallurgy can satisfactorily identify constituents without considerable experience in the examination of alloys containing known constituents. The desirability for all who are interested in the metallography of aluminum to prepare a set of known comparison standards cannot be over emphasized.

The ever present film of flowed aluminum has already been mentioned. The more careful the polish the smaller is the amount of this undesirable film encountered. Likewise, the harder the alloy, that is, the greater the percentage of alloying constituent, the less the surface flow. The etching time required for a given action depends to a certain extent upon the amount of surface flow that has been produced, and the individual operator may find it necessary to slightly vary the time of the etch from that recommended by the authors. In these experiments, great care has been used in the preparation of the specimens and the surface flow has been reduced to a minimum. The polishing procedure has been similar to that previously described in detail[1] and more briefly outlined in the revision of Methods E5.

The etching time and the concentration and temperature have been carefully controlled as designated.

UNETCHED APPEARANCE OF CONSTITUENTS

The first separation of constituents should be based on their color and form in the unetched condition. For judging the color a 5-ampere carbon arc was used with Eastman neutral tint filters, reducing the intensity to 0.5 per cent, and in addition, an Eastman Filter No. 78-A, which has a bluish tint and converts the light of the carbon arc to approximately daylight quality. It is believed that any white light will give approximately the same colors, although the bluish tint aids appreciably in differentiating between constituents. An examination with 4-mm. objective and 10X eyepiece, giving a magnification of approximately 500 diameters, is recommended in all of this work.

Particles of free silicon are readily recognized in the unetched condition and the only effect of the etching reagents described has been to slightly lighten the color which decreases the contrast between it and the purplish-gray constituents. Mg_2Si, under very delicate polishing, approaches the color of silicon, but its tendency to tarnish an iridescent blue, especially when in smaller particles, and its action under the several etching reagents noted makes the identification simple. $CuAl_2$ is readily distinguished from Si and Mg_2Si and, contrary to statements in the literature, is not likely to be confused with the gray constituents. Consequently an etch for coloring $CuAl_2$ is not necessary, although, if properly carried out, the 25-per-cent HNO_3 etch at 70° C. is a satisfactory confirmatory etch. Swabbing with 0.5-per-cent HF for 15 seconds reveals $CuAl_2$ as clear, nearly colorless, sharply outlined particles, and thus increases the contrast between it and other constituents.

THE GRAY CONSTITUENTS

The identification of the gray constituents is a much more difficult matter. When several of these occur in the same field of the

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[1] E. H. Dix, Jr., "Polishing Aluminum and Its Alloys for Microscopic Examination," *Chemical and Metallurgical Engineering*, Vol. 27, p. 1217 (1922).

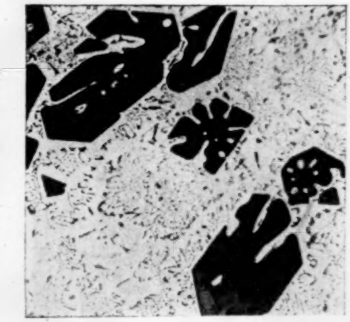

Fig. 8.—Aluminum-Magnesium-Silicon Alloy (No. 19, Mg. 20.15 Per Cent, Si 11.88 Per Cent). Chill Cast in Cold Graphite Mold. Primary and Eutectic Mg_2Si. Unetched. (×500, Reduced One-half in Reproduction.)

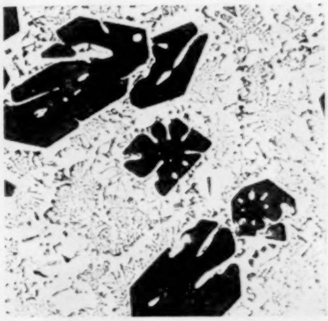

Fig. 9.—Same Section, Showing How Etching Has Removed the Tarnish from the Mg_2Si Particles and Brought out the Eutectic. Etched 0.5-per-cent HF. (×500, Reduced One-half in Reproduction.)

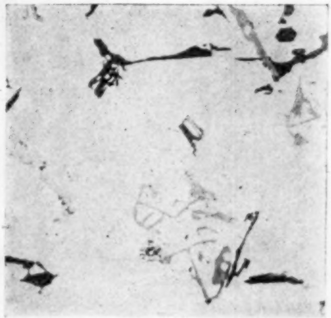

Fig. 10.—Aluminum-Copper-Silicon-Iron Alloy, Sand Cast (Analysis, Cu 6.54 Per Cent, Si 4.59 Per Cent, Fe 1.57 Per Cent). Shows Varying Tones of $CuAl_2$, Light; Silicon, Dark; Iron Constituents, Half-tone. Unetched. (×500, Reduced One-half in Reproduction.)

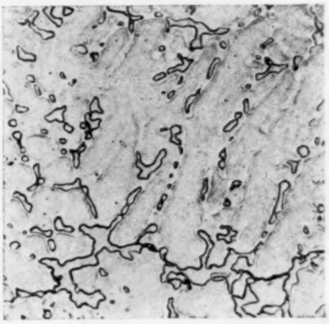

Fig. 11.—Aluminum-Magnesium Alloy Chill Cast in Cold Graphite Mold (No. 6). Mg_2Al_3 and Cored Matrix. Etched 10-per-cent NaOH. (×500, Reduced One-half in Reproduction.)

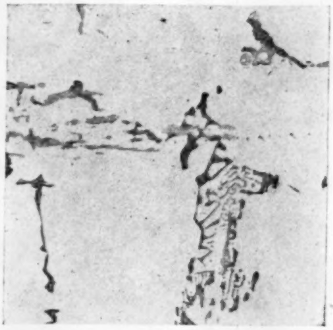

Fig. 12.—Aluminum-Copper-Nickel Alloy, Slowly Cooled Ingot (No. 10, Cu 4.06 Per Cent, Ni 2.01 Per Cent). Shows Three Constituents, $NiAl_3$, Light, Faint; $CuAl_2$, Light; Cu-Ni Constituent, Darker. Unetched. (×500, Reduced One-half in Reproduction.)

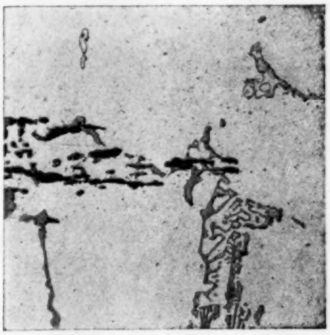

Fig. 13.—Same Field, Showing $NiAl_3$ Colored Brown; $CuAl_2$, Light and Outlined; Cu-Ni Constituent, Darker, Outlined.

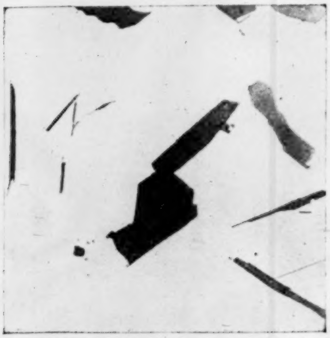

FIG. 14.—ALUMINUM-IRON ALLOY (NO. 3, Fe 7.07 PER CENT). SLOWLY COOLED INGOT SHOWING EFFECT OF ETCH ON $FeAl_3$ PARTICLES. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

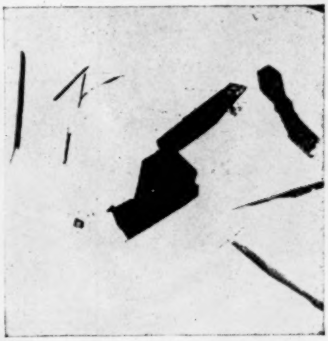

FIG. 15.—SAME FIELD AS ILLUSTRATED IN FIG. 14 FURTHER ETCHED WITH 20-PER-CENT H_2SO_4. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

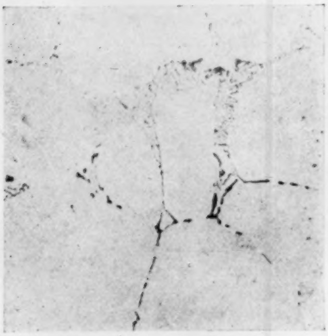

FIG. 16.—ALUMINUM-IRON-SILICON ALLOY (NO. 11, Fe 0.74 PER CENT, Si 0.34 PER CENT) CHILL CAST NOTCH BAR SHOWING Fe-Si IN "CHINESE SCRIPT." UNETCHED. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

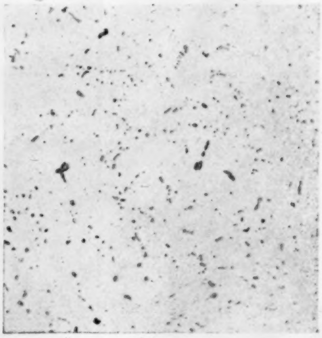

FIG. 17.—ALUMINUM-IRON-SILICON ALLOY (NO. 12, Fe 1.02 PER CENT, Si 0.42 PER CENT). CHILL CAST AND ANNEALED. CONTRAST BETWEEN $FeAl_3$ AND Fe-Si OBTAINED BY ETCHING. ETCHED 25-PER-CENT HNO_3. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

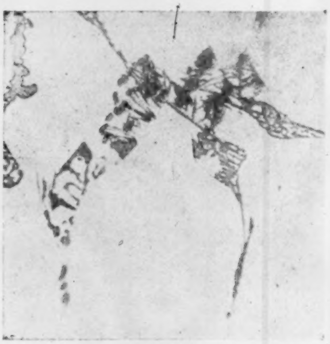

FIG. 18.—ALUMINUM-COPPER-IRON ALLOY (NO. 8, Cu 3.89 PER CENT, Fe 0.50 PER CENT). SLOWLY COOLED INGOT, ANNEALED. $FeAl_3$, Fe-Cu, AND $CuAl_2$. UNETCHED. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

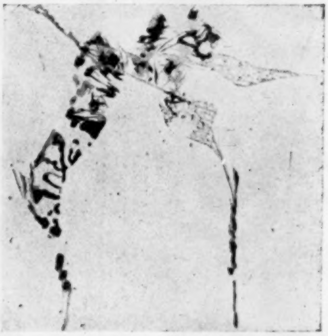

FIG. 19.—SAME FIELD AS SHOWN IN FIG. 18. $FeAl_3$, BLACK; Fe-Cu, LIGHT; AND $CuAl_2$, LIGHT LACEWORK. ETCHED 20-PER-CENT H_2SO_4. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

microscope it is comparatively easy to note distinctions in color contrast between such constituents as $MnAl_3$ and Mn-Si which is generally associated as a lacework bordering on $MnAl_3$. $MnAl_3$ is readily differentiated from most of the other gray constituents because of its flat, gray color. However, such differentiations are very unsatisfactory because the eye cannot carry the tone in going from one part to another part of the same specimen. In the scheme of Fig. 7, etching with 0.5-per-cent HF 15-second swab has been used as a first separation. Four of the constituents are attacked or colored by this etch. These are $NiAl_3$ and Fe-Mn, Mn-Si and Cu-Fe-Mn. The effect of this etch is to roughly divide these constituents into two groups. The Fe-Mn constituent is colored a characteristic smooth brown and the $NiAl_3$ constituent a blue to brown. The other two constituents are roughened and darkened and the Cu-Fe-Mn constituent colored a brown to black. In order to improve the separation between these two groups, etching with 20-per-cent H_2SO_4 at 70° C. —30-second quench—is recommended. The differentiation between the Mn-Si and Cu-Fe-Mn constituents has not been satisfactorily accomplished and there is a further complication when iron, manganese and silicon are present in the same alloy. There is evidence that the Mn-Si constituent is affected by the presence of iron. These matters must be left for later research. In a great many cases the composition of the alloy will furnish this differentiation. Of the five constituents which are not attacked by the 15-second swab with the 0.5-per-cent HF a separation is accomplished by repolishing and etching with 20-per-cent H_2SO_4 at 70° C., 30-second quench. The constituents are thus divided into two groups, $FeAl_3$ and Fe-Si, which are attacked or colored and $MnAl_3$, Fe-Cu and Cu-Ni, which are not attacked or colored. The contrast between the constituents $FeAl_3$ and Fe-Si is greatly increased by etching with 25-per-cent HNO_3 at 70° C. for 40 seconds and quenching. The action of this etch is illustrated in Fig. 17, in which some half dozen particles appear darker and more clearly outlined than the balance. The small black specks are defects due to the polishing and etching. However, this method of etching is hardly satisfactory for differentiating between these two iron constituents when they occur in different specimens, and this subject is being investigated. Two sodium hydroxide etches are recommended for distinguishing the three constituents, $MnAl_3$, Fe-Cu and Cu-Ni, and it is believed that this is satisfactorily accomplished, as indicated in Fig. 7. Etching by swabbing for 10 seconds with 1-per-cent NaOH is indicated as coloring $MnAl_3$ blue to brown. However, this etch is not sufficiently severe to do more than give a spotted, uneven coloring. It has been chosen so that the Fe-Cu will be left unattacked and thus distinguished from $MnAl_3$.

A word of precaution regarding the repolishing as indicated in Fig. 7 should be given. Very confusing and misleading results may be obtained if the effect of the previous etch is not entirely removed. To be safe, it is desirable to go back to the file.

Figures 8 and 9 illustrate both the primary and eutectic particles of Mg_2Si in the unetched condition and after swabbing with 0.5-per-cent HF for 15 seconds. The tarnish on the primary particles of Mg_2Si in Fig. 8 is very typical of the appearance of this constituent as ordinarily polished. The etching has removed this tarnish from the surface of the particles and has revealed the eutectic structure much more clearly. Fig. 10 shows an unetched structure containing silicon, the dark constituent; $CuAl_2$, light, rounded particles; and gray, iron constituents, occurring in script formation and in an elongated plate containing an oval hole in the upper righthand corner. The contrast between the iron constituents and the silicon is very marked and there should be no difficulty in distinguishing the $CuAl_2$ from the iron constituents. However, etching with 0.5-per-cent HF heightens the contrast between $CuAl_2$ and the iron constituents but reduces the contrast between the silicon and the iron constituents.

The compound Mg_2Al_3 is also quite distinctive, since it is much softer than the other constituents and is only faintly revealed in the unetched specimen. This constituent, after etching with 10-per-cent NaOH at 70° C. for 5 seconds, is beautifully revealed in Fig. 11. A grain boundary may be traced along the lower edge of the micrograph and the cored nature of the matrix is well revealed.

$NiAl_3$, $CuAl_2$, and the Cu-Ni constituent are illustrated in Figs. 12 and 13. A comparison of the etched and unetched structures will reveal these three constituents. The 0.5-per-cent HF has colored the particles of $NiAl_3$ brown and has reduced the contrast between the $CuAl_2$ and the Cu-Ni constituent. However, the differentiation between these two is readily apparent in the unetched structure.

The characteristic appearance of $FeAl_3$ after swabbing for 15 seconds with 0.5-per-cent HF and etching for 30 seconds in 20-per-cent H_2SO_4 at 70° C. followed by quenching, is illustrated by Figs. 14 and 15. The only effect of the 0.5-per-cent HF etch has been to more clearly outline the constituents and to reveal cracks extending through the particles. However, the attack of the 20-per-cent H_2SO_4 has been rather severe and the constituent has been roughened and partially dissolved in such a manner as to make the constituent appear black. Figure 16 illustrates the characteristic formation of the Fe-Si constit-

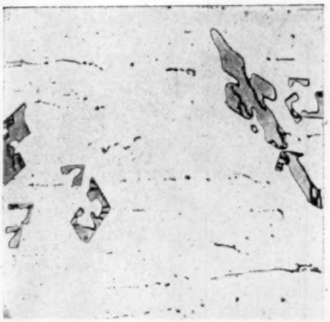

FIG. 20.—ALUMINUM-MANGANESE ALLOY (NO. 5, MN 2.96 PER CENT). CHILL CAST IN GRAPHITE MOLD. CHARACTERISTIC $MnAl_3$ FORMS. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

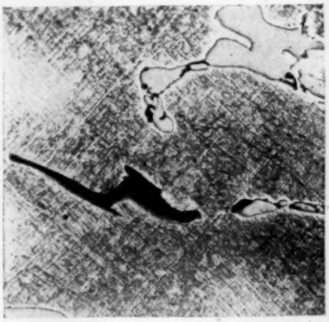

FIG. 21.—STRONG ALLOY INGOT, SLOWLY COOLED (NO. 27, NOT ANNEALED). Mg_2Si, DARK ASSOCIATED WITH $CuAl_2$, LIGHT. THE LARGE, BLACK PARTICLE IS THE Cu-Fe-Mn CONSTITUENT. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

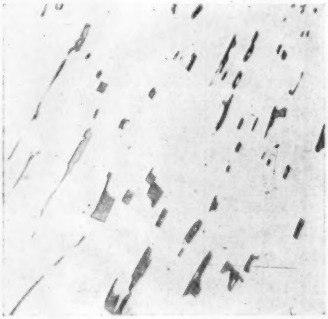

FIG. 22.—ALUMINUM-IRON-MANGANESE ALLOY. (ANALYSIS, FE 0.63 PER CENT, MN 0.77 PER CENT.) SLOWLY COOLED INGOT SHOWING Fe-Mn CONSTITUENT. UNETCHED. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

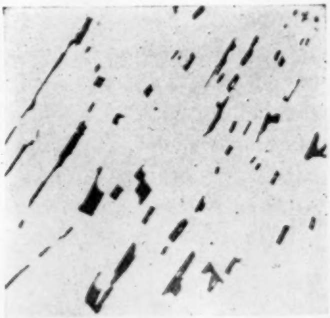

FIG. 23.—SAME FIELD AS SHOWN IN FIG. 22, ILLUSTRATING THE EFFECT OF ETCHING ON THE Fe-Mn CONSTITUENT. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

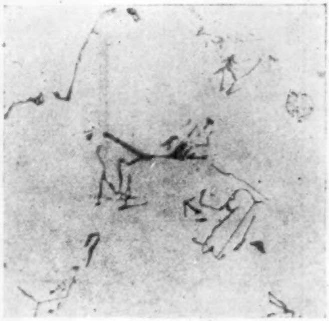

FIG. 24.—ALUMINUM-MANGANESE-SILICON ALLOY. (NO. 17, MN 2.12 PER CENT, SI 1.85 PER CENT.) CHILL CAST IN GRAPHITE MOLD. SHOWS $MnAl_3$ AND Mn-Si CONSTITUENT. UNETCHED. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

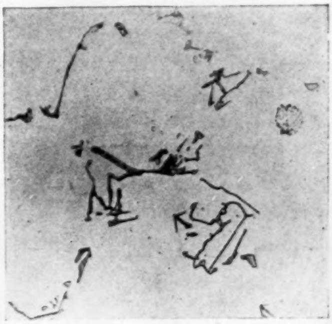

FIG. 25.—SAME FIELD AS SHOWN IN FIG. 24, ILLUSTRATING EFFECT OF ETCH. Mn-Si, DARK; $MnAl_3$, LIGHT. ETCHED 0.5-PER-CENT HF. (×500, REDUCED ONE-HALF IN REPRODUCTION.)

uent as it occurs in cast alloys. Two iron constituents in a similar alloy after thorough annealing are shown in Fig. 17.

Figures 18 and 19 illustrate the appearance of three constituents in an aluminum-copper-iron alloy containing 3.89 per cent of copper and 0.50 per cent of iron. Etching for 30 seconds in 20-per-cent H_2SO_4 at 70° C. followed by quenching has blackened the $FeAl_3$. Neither of the other two constituents has been affected. In these micrographs the $CuAl_2$ appears as a lacework, whereas the Fe-Cu constituent appears as needles and irregular plates which are slightly darker than $CuAl_2$ and are readily distinguished under the microscope. The characteristic appearance of the $MnAl_3$ constituent after swabbing with 0.5-per-cent HF for 15 seconds is illustrated in Fig. 20. In Fig. 21 three constituents, in an alloy of the duralumin type containing 3.52 per cent of copper, iron 0.48 per cent, silicon 0.42 per cent, manganese 0.55 per cent, and magnesium 0.46 per cent, are illustrated. The specimen has been etched by swabbing with 0.5-per-cent HF for 15 seconds. This has left the $CuAl_2$ characteristically clear and definitely outlined, has stained the aluminum solid solution matrix, revealing numerous scratches, and has colored a large particle of constituent an uneven brown. This particle is the Cu-Fe-Mn constituent. Particles of Mg_2Si are associated with the $CuAl_2$ in a characteristic manner and after this etching appear a bright blue, but photograph black.

The characteristic appearance of the Fe-Mn constituent unetched and after swabbing for 15 seconds with 0.5-per-cent HF is illustrated in Figs. 22 and 23. In the unetched condition, as shown by Fig. 22, this constituent cannot be differentiated from $MnAl_3$ either by form or color, but the HF etch colors the particles an even brown, as illustrated in Fig. 23, which are readily differentiated from $MnAl_3$ as shown in Fig. 20. Figs. 24 and 25 illustrate the constituents $MnAl_3$ and Mn-Si. In the micrograph of the unetched structure, Fig. 24, the darker lacework particles of the Mn-Si constituent may be seen. These have been roughened and darkened by swabbing for 15 seconds with 0.5-per-cent HF, whereas the $MnAl_3$ particles have the same characteristic appearance illustrated in Fig. 20.

Acknowledgement.—The authors desire to express their appreciation to Mr. G. W. Wilcox for his efficient aid in preparing specimens and in the development of negatives and prints, and to Mr. H. V. Churchill under whose direction the chemical analyses were made.

ALUMINUM CASTING ALLOYS:

THE BRITISH ENGINEERING STANDARDS ASSOCIATION SPECIFICATIONS REVIEWED

By George Mortimer[1]

Synopsis

This paper briefly discusses the aluminum casting alloys covered by the British Engineering Standards Association specifications.

The methods specified for checking their quality on routine output are reviewed and compared with current methods in America. It is felt by the author that in few cases in routine work can any test bar represent the strength of a casting. If this is the case it seems preferable to concentrate on an accurate indication of the quality of material going into that casting. It is shown that both a sand-cast coupon integral with the casting and the B.E.S.A. chill-cast separate bar as at present specified involve too many variable factors to give a reliable indication of the quality of the melt.

One method is described which eliminates variables of practical import in connection with a separate chill-cast bar. The test results obtained may be far higher than those likely to be found in the sand-casting the bar represents. The value of the method, however, lies in the consistency of results obtained from the same melt, and the marked difference in results obtained by any change in material or melting practice.

Alloy Compositions

With the exception of castings for marine work, practically all of the requirements of the British Isles in aluminum castings are represented by the British Engineering Standards Association specifications for light casting alloys. The requirements of these specifications are briefly given in Table I.

Alloy 2-L-5:

The best known and most generally used alloy in the United Kingdom is alloy 2-L-5. Thirty years ago when aluminum casting was in its infancy it was found that, although the pure metal could be cast, its properties were unlikely to attract engineers: tensile strength was relatively low, and machining properties left much to be desired. Addition of zinc, a cheap and convenient metal which could be added direct to the pot, considerably improved matters. The tensile strength could be doubled and machining was excellent,

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[1] Managing Director, William Mills Foundry, Birmingham, England.

so aluminum-zinc alloys became popular in England and on the Continent. The amount of zinc added varied from 8 to 20 per cent, and gradually became fixed at a compromise value of some 15 per cent.

Zinc has one drawback in that for a wide range of temperatures after solidification all aluminum-zinc alloys are weak. They are tender in the mold, and this just at a time when considerable contraction is taking place. In other words, zinc alloys are "hot-short," a prolific cause of cracked castings.

TABLE I.—REQUIREMENTS FOR LIGHT CASTING ALLOYS, ABSTRACTED FROM SPECIFICATIONS OF BRITISH ENGINEERING STANDARDS ASSOCIATION.

Alloy Specification Number	Specified Chemical Composition	Impurities					Minimum Specified Tensile Properties		Average Tensile Properties Obtained		Specific Gravity
		Iron, min., per cent	Silicon, min., per cent	Lead, min., per cent	Zinc, min., per cent	Tin, min., per cent	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	
3-L-11	Copper, not less than 6 per cent nor more than 8 per cent. Tin, may be present up to 1 per cent. Aluminum, the remainder.	0.80	0.70	0.10	0.10	....	20 160	3	20 160 to 29 640	4 to 6	2.88
2-L-8	Copper, not less than 11 per cent nor more than 13 per cent. Aluminum, the remainder.	0.80	0.70	0.10	0.10	....	20 160	..	24 640 to 26 880	1 to 2	2.90
2-L-5	Zinc, not less than 12.5 per cent nor more than 14.5 per cent. Copper, not less than 2.5 per cent nor more than 3.0 per cent. Aluminum, the remainder.	0.80	0.70	0.10	....	....	24 640	3	31 360 to 35 840	6 to 10	3.00
L-24	Copper, not less than 3.5 per cent nor more than 4.5 per cent. Nickel, not less than 1.8 per cent nor more than 2.3 per cent. Magnesium, not less than 1.2 per cent nor more than 1.7 per cent. Aluminum, the remainder.	0.80	0.70	0.10	0.10	0.10	As cast, 24 640 Heat-treated (no specification yet issued)	.. ..	As cast 20 160 to 24 640 Heat-treated, 38 080 to 49 280	1 to 2 3 to 6	2.85

NOTE.—The aluminum used for the above alloys shall be virgin metal. No scrap shall be used other than that derived from the maker's own manufacture under this specification. The copper shall assay not less than 99.3 per cent. The nickel shall assay not less than 99 per cent. The zinc shall assay not less than 99.5 per cent.

Test bars shall be 1 in. in diameter and 7 to 9 in. long, cast in iron chills which have been heated before they are filled. The bottom of the chill shall be closed with a sand or clay plug.

Substituting some 3 per cent of copper for an equivalent amount of zinc gives an alloy no less strong and at least as easy to machine, while the tendency to hot-shortness is definitely reduced. This alloy, 2-L-5, has been in constant use for at least twenty years in England to the exclusion of most others for such parts as automobile crankcases and gear-boxes and highly stressed parts of a similar nature on airplane engines.

Alloy 2-L-5 is an easy and fluid alloy to work with in the foundry, reasonably cheap, strong and stable in all climates. In applying it to engineering, however, one fact has to be borne in mind. The aluminum alloys containing zinc are weak at high temperatures. Accordingly, alloy 2-L-5 is never used for automobile pistons, and should not be applied to any job calling for a similar range of temperature.

Alloy 3-L-11:

Castings required to withstand high temperatures are better cast in a straight copper alloy, the best known of which in England is 3-L-11.

Aluminum-copper alloys were little used until the World War, when serious efforts were made to find a more satisfactory alloy for aircraft pistons than was afforded by the zinc group. One of the more successful was an alloy consisting of 7 per cent of copper, 1 per cent of zinc, 1 per cent of tin, and the balance commercial aluminum. This alloy was designated L-11. Later it was found that even the small percentage of zinc contained in this mixture was detrimental at high temperatures, and so the zinc was eliminated, and the alloy became 2-L-11. Later still the tin content was made optional; it is not quite clear why it was ever included, except possibly to impart a slightly lower crystallization shrinkage. The final specification, 3-L-11, is now identical with No. 12, an alloy which has been tried out in practice to quite the same extent as 2-L-5, and which has the further advantage in being better able to withstand high temperatures.

Alloy 2-L-8:

The higher the copper content, the lower the total shrinkage, and this may be of service in the manufacture of difficult castings. Within limits, the more copper used the less the tendency to general porosity, a point of value in the production of castings designed to withstand hydraulic pressures. Finally, a higher copper content gives a relatively hard, well-finished machined surface, very suitable for such parts as automobile pistons.

The addition of copper, however, must be reasonably controlled, for it involves an increasing brittleness in the casting, together with an increase in weight disproportionate to any gain in strength.

A specification was issued covering an alloy with a copper content of 12 per cent known as alloy 2-L-8, and this alloy is used for the great majority of automobile pistons cast in England, whether in sand or in permanent molds.

Alloy L-24:

The alloys so far described represent the repertory of the majority of founders in the British Isles, and they meet the needs of about 80 per cent of the requirements in the country for aluminum alloy castings. It would not be claimed that any one of them represented the last word in light alloy founding practice, but they have the great advantage of facility of working, of being well understood by designers and founders, and of a very long record of reliable service under widely varying conditions of design, duty and climate.

There remains alloy L-24, probably more widely known as "Y" alloy, an alloy of the duralumin class which has the advantage of lending itself readily to the process of casting. This alloy is primarily the product of the National Physical Laboratory in its war-time attempt to effect that most difficult of compromises: a piston alloy giving the maximum strength at working temperatures consistent with high thermal conductivity and good frictional properties. The history of its development is detailed in the Eleventh Report of the Alloys Research Committee. The main point of interest in connection with the alloy is its susceptibility to heat-treatment, and the excellent tensile and fatigue properties exhibited by a casting of this alloy when properly cast and heat treated. For a long time there was a reluctance to adopt an alloy which cost more, the foundry treatment of which was more intricate, expensive and uncertain in results than in the case of alloys in current use.

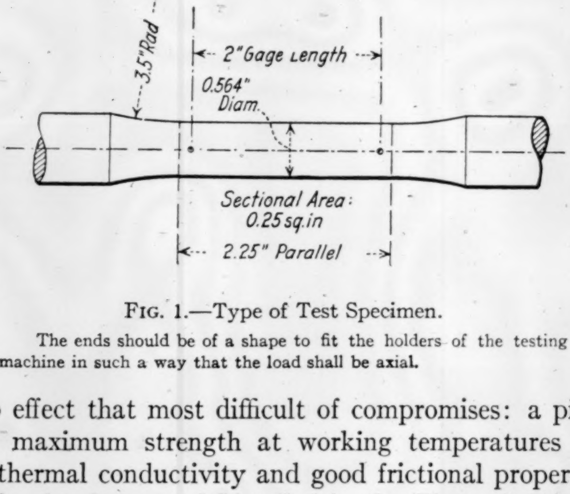

FIG. 1.—Type of Test Specimen.

The ends should be of a shape to fit the holders of the testing machine in such a way that the load shall be axial.

With growing knowledge of essentials of heat treatment of light alloys generally, there has been a decided reaction in favor of L-24. Its properties have been found of constructive value in light alloy engineering and it has tended to open up new fields for aluminum castings. In the author's experience the physical properties of heat-treated castings of this material show a marked advance over the other alloys, while in addition the material may be said to possess a toughness and reliability in service which is difficult to convey by means of specification figures. Whenever large Diesel engine pistons have to be made to withstand abnormal temperature conditions, or any aluminum casting is needed for some special high-duty service, "Y" alloy is specified in nine cases out of ten, although the founder would prefer to use almost any other, if he had the same confidence in the results.

SPECIFICATION TEST REQUIREMENT

Castings are primarily accepted or rejected in the rough on analysis and on the results of tension tests of a standard bar. The requirements for the bars, their manufacture and dimensions, are probably of interest and are therefore quoted in full below:

TENSION TEST

Test specimens shall be turned to the dimensions of British Standard Test-Piece C. (A suitable test piece is shown in Fig. 1.) When tested they shall give:

Ultimate tensile strength..........not less than .... tons per sq. in.
Elongation......................not less than .... per cent

The test pieces shall not be annealed, hammered or otherwise treated before they are tested.

At least one bar shall be cast to represent each large casting. For small castings the number of bars shall be agreed upon by the authorized inspector [generally arranged to represent each heat (author's note)] so that all the alloy used is tested. The bars shall be cast from the same ladle as the castings and shall be poured first.

The bars shall be 1 in. in diameter and from 7 to 9 in. long. They shall be cast in iron chills which have been heated before they are filled. The bottom of the chill shall be closed with a sand or clay plug, not with a metal end.

The British specifications depart from accepted American practice. The latter aims primarily at indicating to the engineer something of the properties he will find in the casting; British practice aims entirely at a standard test which will reflect the quality of the material going into that casting, and it is contended that the integral sand-cast coupon cannot reliably fulfill that function.

As a founder of aluminum alloys, with experience of both systems under routine conditions, the author offers the following observations.

The American practice has a great advantage in simplicity and economy, in the avoidance of special molds and extra handling; there is no machining to more or less fine limits or delay of castings while this is being carried out, and no elaborate system of stamping and checking to ensure correct correlation of bars with their castings. Finally, on relatively simple work the American design of coupon can reflect very fairly the properties likely to be found in the casting it is cut from.

On the other hand, in the great majority of castings the properties tend to vary throughout the different sections. There are sections which, owing to their position, cannot either be adequately fed or chilled. The effect is a wide variation in tensile strength and elongation in different parts in a casting, amounting in observed instances to the figures called for by the specification itself.

This is generally recognized, and a designer of experience therefore uses a material he knows, and allows a good margin of safety.

The sand-cast integral coupon may not indicate the quality of the material or the standard of melting practice. It may rather reflect the individual skill of the foundryman in its placing, molding and feeding. The coupon may be attached to thick or thin sections, molding in green or dry sand, cast upright or horizontal, and in fact be liable to most of those variations which affect the results throughout an ordinary casting. Further unknown quantities are introduced by surface irregularities, the effect of the skin, and of a relatively small internal defect, perhaps normal to sand castings but bearing an undue proportion to the sectional area of the small bar.

The British Standards authorities, therefore, contend that an integral bar does not necessarily give much information about the casting and specify a bar cast separately, under conditions calculated to give uniform results.

On the other hand the British specifications sweep away a host of unknowns only to introduce new ones as follows:

1. Temperature of metal poured.
2. Temperature of chill mold.
3. Rate of pouring.
4. Thickness of chill.
5. Chill design and dressing, in relation to freedom of contraction in the bar.

Any one of these factors can materially affect the results on a series of bars cast from the same pot of metal, and not one of them is covered in the specifications. Obviously, therefore, the system as it stands falls short of definitely reflecting the quality of the metal poured.

All that would appear to be necessary would be the provision fo having the iron mold near the job, which the molder would fill up before he poured his casting. If the above points are disregarded, however, far more widely varying results may be obtained than are ever experienced with the sand-cast coupon. Frequently a costly and difficult casting is irrevocably rejected owing to complete failure of its test bars to give specification figures, while its neighbor, cast at the same time from the same pot of metal, is accepted on figures correspondingly high. While that state of affairs is possible, the separate, chill-cast bar cannot be regarded as an accurate gage of the quality of material. If it cannot be so regarded, then there seems little excuse for departing from the essential simplicity of the integral cast bar, cast to size, without machining.

PROCEDURE FOR TESTING FOR PLANT CONTROL—A METHOD DESIGNED TO ELIMINATE VARIABLES

The author is inclined, however, to favor the principle of accurately testing the quality of the material, as distinct from indicating the strength of a casting. With this in view the specification requirements have been carried a little further in the foundry he is associated with, and the following procedure has been adopted as the result of a long series of practical tests.

The crucible *en route* to the mold passes first to a small gas-fired furnace equipped with a pyrometer, and metal for the required number of bars is poured into a pot in that furnace. If it is above the standard temperature it is allowed to cool down to it; if below, heat is applied until the temperature is right. The standard temperature decided on is 730° C. for alloy 3-L-11, 715° C. for alloy 2-L-5, and 745° C. for "Y" alloy (L-24). The chill molds are arranged in racks alongside this heating unit, and are fixed at an angle slightly inclined from the vertical.

Beneath each chill mold is a gas burner which maintains the chill dry and between the temperatures of 100 and 150° C. The difference in physical properties in bars poured into chills at any temperature within this range appears to be negligible. The chills are of cast iron, machined within, and have an even section of ½ in. They are split vertically and the two halves are dowelled together very accurately to avoid the formation of any sort of flash which could check free contraction of the bar. Before each bar is cast, the chill is dressed with graphite to prevent as far as possible any friction between the bar and mold when the former is contracting. In addition, the upper half of the mold is fitted with a handle, and is removed immediately the bar has set in the interest of free contraction. These precautions are the result of much experimental work on the part of N. F. Budgen, the company's chief metallurgist, who found that this factor had an important influence on both the tensile strength and elongation results obtained.

The apparatus used is illustrated in Fig. 2. The temperature is fixed for the metal by the furnace, at whatever temperature the casting has to be poured. The thickness of chill is standard for all jobs, and the temperature is fixed within a limited range. The rate of pouring is reasonably fixed by the employment of a ladle drilled with a small hole instead of having the usual lip. The pouring is so slow that the lower sections are almost solid before the mold is half full, and this takes care of crystallization shrinkage without the necessity for a header, which might check the subsequent solid contraction. The latter is free owing to the absence of flashes or headers, and the presence of a smooth graphite surface.

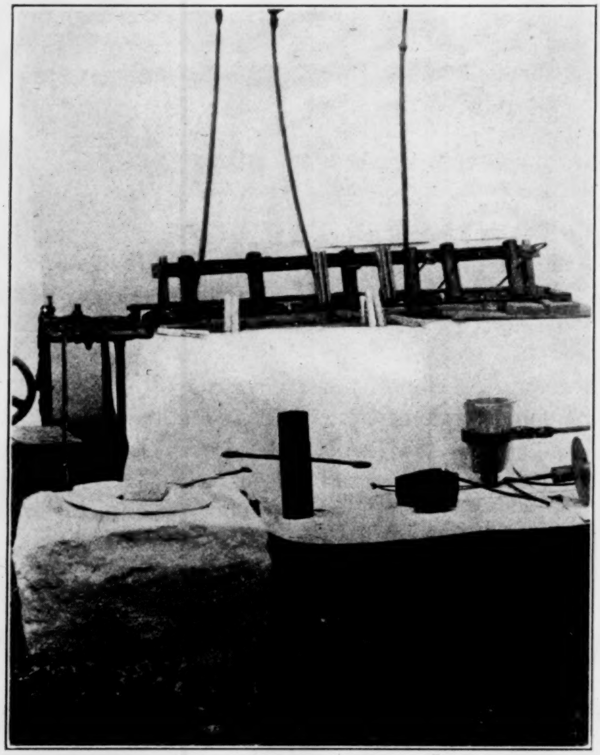

FIG. 2.—Apparatus for Casting Test Bars.

The net results of these refinements are a striking improvement in test bar figures, and the fact that the values obtained represent the actual strength of the casting less than ever. The test bar results do begin to reflect the actual quality of the melt. Bars from the same melt, whether giving high or low figures, do at any rate check up within reasonable limits.

This procedure is well worth while from the founder's point of view. Taken in conjunction with daily analyses and pyrometer charts, test bar results obtained under these fixed conditions constitute a very useful barometer as to the state of affairs in the buying department and in the melting shop. Incidentally, it is a barometer with a large scale dial. If alloy 3-L-11 (No. 12) is taken, for instance, the specification calls for a tensile strength of 9 tons (20,160 lb. per sq. in.) with 3 per cent elongation. Whatever the grade of metal used, when using the integral cast bar, the range of properties is limited. When using a chill mold the properties vary widely according to the grade of material and the melting practice used, a point of great value to the founder who aims at high results.

This is illustrated by Table I. Taking alloy 2-L-5, (aluminum 84 per cent, zinc 13 per cent, copper 3 per cent) it is seen that although the specification calls only for 11 tons (24,640 lb. per sq. in.) with 3 per cent elongation, the results obtained under routine conditions vary between 14 and 16 tons (31,000 and 35,000 lb. per sq. in.) with from 6 to 10 per cent elongation.

Six bars were put through routine practice for the purposes of this paper, and the results are given herewith.

TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION IN 2 IN., PER CENT
38 980	9.0
38 240	8.0
37 860	10.0
36 830	8.0
39 870	10.0
39 444	10.0

The lesson to be derived from the above figures, apart from the high results possible under ideal conditions, is the consistency of the figures. This consistency is found throughout the whole range of desirable or undesirable material, from 22,400 lb., with 2 per cent elongation, up to 38,080 lb. with 10 per cent elongation. So long as bars are cast from the same melt, they will closely tally in their tensile results and reflect the quality of material going into the casting.

This is a point of real value to the founder of aluminum alloys, who has to contend with at least two sources of contamination which at present cannot well be determined by routine analysis: the content of alumina and the content of dissolved gases. He may buy the best metal, but these two factors may upset all his calculations owing to carelessness in melting. They cannot be detected by any analytical process known to the author, but their effect on test bars cast as described is immediate and quite definitely marked. When his test bar figures fall off, he can feel reasonably confident that material or melting practice needs attention, simply because the chief variables associated with a sand-cast coupon, or a chill-cast separate bar made under unspecified conditions, are absent.

Summing up, it will always be very difficult to gage the strength of a casting from a bar cast integral with it. If in view of this it is decided that a gage of the quality of the material going into that casting is the more satisfactory test, then the test should be so specified in detail as to eliminate all variables of practical import, other than those directly associated with quality of material. It would appear that both the integral sand-cast coupon and the chill-cast bar as specified by the B.E.S.A. standards fall short of this ideal.

DISCUSSION

THE CHAIRMAN (*Mr. William Campbell*[1]).—Gentlemen, when I read this paper over it rather appealed to me, not from the viewpoint of the aluminum alloys, because I do not know much about them, but from the viewpoint of the bronzes. One of our bronzes, gunmetal, is specified in our standards at 30,000 lb. per sq. in. tensile strength and 14 per cent elongation; yet if the test bar barely meets that, it represents pretty poor metal. We have had to put the specifications so low simply on account of sloppy foundry practice. It is so easy to take gunmetal ingots and ruin them in the foundry and then say "Oh well, this alloy we have is absolutely worthless." That is the excuse one hears time and time again. **Mr. Campbell.**

I think there is a great deal to Mr. Mortimer's paper and I should be glad to have Mr. Jeffries discuss it.

MR. ZAY JEFFRIES.[2]—It is fortunate that the author of the paper has so clearly described the method of manufacture of his test bars. Take, for example, the alloy 3-L-11; our specifications for the corresponding alloy No. 12, cast in sand, are 18,000 lb. per sq. in. minimum tensile strength and 1 per cent elongation in 2 in. It is not possible, by any method of which I have knowledge, to cast No. 12 alloy in sand with anything like the physical properties given by Mr. Mortimer in his paper, namely, 20,160 to 29,640 lb. per sq. in. tensile strength and 4 to 6 per cent elongation, no matter what kind of metal is used, no matter what kind of sand is used, or what the pouring temperature may be. As a matter of uniformity, aluminum sand castings are notably more uniform than aluminum chilled castings; the chilling itself introduces a variable in an aluminum alloy which is greater than most of the other variables encountered in the casting of the several alloys. **Mr. Jeffries.**

Alloy 2-L-5 has, in a sand casting freshly cast, somewhere in the neighborhood of 25,000 to 26,000 lb. per sq. in. tensile strength and about 2.5 per cent elongation; on aging at room temperature for a year, the tensile strength may rise to a value of 30,000 lb. per sq. in. and the elongation may drop to less than 1 per cent. It has good machining properties and is used for the bulk of crank-case castings

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[1] Metallurgist, New York Navy Yard, and Howe Professor in Metallurgy, School of Mines, Columbia University, New York City.

[2] Consulting Metallurgist, Aluminum Co. of America, Cleveland, Ohio.

Mr. Jeffries. and the like in England largely for that reason and for its relative cheapness. Alloy 2-L-5 is, however, used very little in chilled castings. The largest chilled castings manufacturers in England use the alloys similar to 3-L-11 and 2-L-8. The tensile properties of a chilled test bar are very much better than the properties of sand castings of the same alloy.

The alloy L-24, commonly known as Y alloy, is more than a casting alloy; it is a forging alloy, a sheet alloy, a sand-casting alloy and a permanent mold casting alloy. When cast in sand, its properties are uninteresting, that is, its tensile properties. When cast in sand and heat-treated, the tensile strength may be 35,000 lb. per sq. in. and the elongation possibly 1.5 per cent. These properties are uninteresting as compared with the properties now obtainable in this country in commercial heat-treated castings. So far as the general versatility of L-24 is concerned, it perhaps has no equal; that is, if we were to select one aluminum alloy for all purposes, chilled castings, wrought metal and sand castings, I think we could select L-24 to advantage. However, I believe there is no single use in which L-24 excels, and in this country, where we have a large production for specific purposes, we naturally turn to the cheaper alloys. It can be readily seen that L-24 is an expensive alloy; it contains 2 per cent of nickel and 1.5 per cent of magnesium, both metals being relatively expensive. For pistons they are using in England an aluminum-copper alloy similar to the one used in this country. We use an aluminum-copper alloy, not because L-24 does not make a good piston, but because it is not economical. It does not make a better piston, so far as our tests show, than the alloy of 10 per cent copper, 0.25 per cent magnesium and 1.25 per cent iron; it makes a piston substantially as good as the piston used in this country. Y alloy has excellent properties at high temperature, yet no aluminum alloy we know of has better high-temperature properties than one containing about 12 per cent copper and 0.75 to 1 per cent manganese, which is cheaper than Y alloy. It should be kept in mind that the figure given for L-24, heat-treated, namely, tensile strength 38,080 to 49,280 lb. per sq. in. and elongation 3 to 6 per cent, is the result of a very special heat treatment of a chill-cast test bar. In chill-cast test bars we have produced values as high as 54,000 lb. per sq. in. tensile strength and 18 per cent elongation, so that, as a peak figure for heat-treated aluminum alloys, the Y alloy is not the best one that can be selected.

I wish to express my appreciation to Mr. Mortimer for sending this paper across the water to us so that we can compare information

Mr. Jeffries.

on these various alloys, because I think it is very helpful. Other people may not be of the same opinion as I am regarding the chilled test bar in evaluating sand castings, but we have tried both ways and we have come to the conclusion that we are better off, both economically and from the standpoint of evaluating the metal, by using sand-cast test bars to evaluate sand castings and chill-cast test bars to evaluate chilled castings.

Mr. Campbell.

THE CHAIRMAN.—I was hoping Mr. Jeffries would tell us something about the relation of the sand-cast test bar to the casting itself. What I wanted to bring out in my earlier remarks was that you never get a lot of dross, dirt, floating cores and so on in the test bar, but you do get them in the casting.

Mr. Jeffries.

MR. JEFFRIES.—The comparison of 88-10-2 alloy with aluminum is not a good one. Sand castings of aluminum themselves vary in properties as compared to the test bars, but sometimes the castings have better physical properties and sometimes poorer, and the controlling thing is the degree of chill. It is not a matter of dross and lack of integrity of the section. The properties of the ½-in. test bar will be closely approximated in a ¼-in. section of a casting and a section less than a ¼-in. in thickness will very often have better physical properties than those of the test bars. The larger sections will have properties in the neighborhood of 70 to 75 per cent of those of the test bar.

Mr. Patch.

MR. N. K. B. PATCH.[1]—I think one of the interesting features of this paper is the fact that the author pointed out that the relationship between the test bar and the casting is simply a relative one and that the test bar really represents the metal itself under standard conditions, rather than the metal in the casting. In other words, the tensile strength of the casting may or may not be that of the bar depending upon the cooling conditions, casting practice and many variables. However, the test bar is something that can be definitely standardized, either chilled, sand-cast or whatever it may be, and it can be laid down on a carefully planned basis, so that we can get a uniform test bar to represent the quality of the metal in the ladle, and the test bar only represents that under any circumstances.

Mr. Jeffries.

MR. JEFFRIES.—I should like to point out that the author of the paper seems to have in mind that our standard practice is the casting of coupons on the casting itself. This is required by some government bureaus, and then only in certain cases, but our standard practice is the pouring of test bars in separate molds with careful control of conditions as outlined by Mr. Mortimer and as suggested by Mr. Patch.

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[1] Secretary, Lumen Bearing Co., Buffalo, N. Y.

Mr. Pannell.

MR. E. V. PANNELL[1] (*closure by letter*).—On behalf of the author I should like to express appreciation for the reception of this paper. As Mr. Jeffries indicates, the chill-cast bar does not represent the strength of the casting, but as Dix and Lyon conclusively proved two years ago,[2] the sand-cast coupon does not represent the strength of the casting either. The object of this paper is to suggest that the chill-cast bar poured under uniform conditions gives the most consistent record of the metal poured in daily routine and so assists foundry control.

In commenting on the L-24 Alloy and comparing it with the typical American mixture for pistons, it will be noted that the latter contains added iron up to 1.25 per cent. This is absolutely contrary to British practice in that iron is believed to be a harmful constituent giving rise to considerable shrinkage with a tendency to brittleness and hard spots. After considerable experimentation, nickel was adopted as a hardening element and the iron content held at the minimum. The L-24 alloy is appreciably lighter than the American piston alloy and the nickel content appears to improve the thermal conductivity as well as giving a very fine finish on machining; in fact the high polish obtainable on grinding is of considerable value in piston and cylinder service.

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[1] Technical Advisor, The British Aluminum Co., Ltd., New York City.

[2] E. H. Dix, Jr., and A. J. Lyon, "Physical Properties of Some Copper-Silicon-Aluminum Alloys when Sand Cast," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 250 (1922).

SOME MECHANICAL PROPERTIES OF DURALUMIN SHEET AS AFFECTED BY HEAT TREATMENT[1]

By Robert J. Anderson[2]

Synopsis

The effects of various annealing, quenching, and aging heat treatments on some mechanical properties of duralumin sheet were examined in the investigation described in this paper. The sheet varied in thickness from No. 10 to No. 30 B. & S. gage. In the annealing heat-treatment experiments, the effects of heating at various temperatures followed by air and furnace cooling were examined. In the quenching and aging heat-treatment experiments, the effects of quenching temperature, time period of aging at the ordinary temperature, temperature of aging for a constant time, time period of aging at a constant elevated temperature, time period of soaking prior to quenching, and different quenching media were examined.

The mechanical tests made on the sheet included tension, hardness, and indentation (cupping) tests. The main results of these tests are presented in graphical form, and the data are discussed briefly. The graphs show suitable heat treatments to yield particular required properties. In the tension testing of duralumin, the interesting phenomenon of slippage is encountered, and the observations made on this are briefly described. The conclusions drawn from the experiments are based on a wide variety of heat treatments and tension tests on about 1000 specimens, with a correspondingly large number of hardness and cupping tests.

Introduction.

This paper gives a summary of the results of a series of tests made to determine the effects of a variety of heat treatments on certain mechanical properties of duralumin sheet. The scope of the test work was comprehensive, and the heat-treatment experiments covered ample range to provide data concerning the effects of a wide variety of annealing, quenching, and aging treatments. The data reported in the present paper were obtained during the course of an extensive investigation concerned with (1) a study of the theoretical aspects of the duralumin phenomenon, (2) a study of the effects of heat treatment on certain mechanical properties of duralumin sheet, and (3) a study of the effects of heat treatment on the microstructure of duralumin sheet. The effects of a wide variety of heat treatments on the mechanical properties (principally tensile properties and hardness) of duralumin sheet have not been investigated fully hitherto, other than by Grard.[1] The author's experiments have been more detailed and extensive than those of Grard, and the data obtained fill several large gaps hitherto existing in the published information.

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[1] Abstract of part of a thesis presented to the Massachusetts Institute of Technology (May, 1925) in partial fulfillment of the requirements for the degree of Doctor of Science.

[2] President, Robert J. Anderson, Inc., Cincinnati, Ohio.

The effects of various heat treatments on the indentation (cupping) properties of duralumin sheet have been studied but little, and the data given in this paper may be of interest in connection with specifications on the drawing and forming qualities of duralumin sheet. Published papers giving the results of precision extensometer tests on duralumin are few, and the present tests should serve to supply information in this direction. Few reported data are available regarding the Brinell hardness of duralumin sheet; in the present investigation the effects of a variety of heat treatments on the Brinell hardness (small-ball machine) of this material have been studied.

Acknowledgments.—The duralumin sheet used in this investigation was rolled by the Baush Machine Tool Co., Springfield, Mass., through the courtesy of Messrs. James T. Johnson, Jr., and R. W. Daniels, and the tension test specimens were machined in part by the Engineering Division of the U. S. Air Service, McCook Field, Dayton, Ohio, through the courtesy of Mr. J. B. Johnson and Lieut. A. J. Lyon, and in part by the General Electric Co., Schenectady, N. Y., through the courtesy of Mr. D. Basch. The experimental work of heat treatment and mechanical testing were carried out in various laboratories of the Massachusetts Institute of Technology, Cambridge, Mass.

MATERIAL EMPLOYED IN THE EXPERIMENTAL WORK

The duralumin employed in this investigation was in the form of sheet of Nos. 10, 14, 18, 22, 26, and 30 B. & S. gage. Nos. 10 and 26 gage were rolled in a high-copper alloy and the other gages in a low-copper alloy. The average chemical compositions of the lots of sheet were as follows:

ELEMENT	HIGH-COPPER ALLOY	LOW-COPPER ALLOY
Copper, per cent	6.05	4.03
Iron, per cent	0.79	0.80
Silicon, per cent	0.20	0.24
Magnesium, per cent	0.46	0.45
Manganese, per cent	0.63	0.59
Chromium, per cent	0.11	0.10
Aluminum (by difference), per cent	91.76	93.79

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[1] C. Grard "L'Aluminium et ses Alliages," Paris, 1920, Berger-Levrault; and translation into English by C. M. and H. C. L. Phillips, New York, 1922, D. Van Nostrand and Co.

The compositions of the two alloys were substantially the same except for the copper content. The low-copper alloy represents regular commercial material.

The alloys were made up from primary first-grade aluminum and the required hardeners and additive metals. Rolling ingots, 3 by 14 by 18 in., were poured in book-type vertical cast-iron molds at about 700° C. The ingots were broken down at 450° C., after pre-heating, to slabs ¼ in. thick, and the sheets were then rolled to gage according to the usual mill practice, using intermediate annealing between passes when required. No cross rolling was done. The sheets were cold finished to gage, the reduction being from 2 to 6 gage numbers in the cold after annealing at 425° C., the amount of reduction depending upon the thickness of the sheet and the tendency exhibited toward cracking. Specimens for tension tests were cut in the direction of rolling.

The bulk of the tests centered around the No. 18 gage low-copper sheet, and the subjoined figures give the mechanical properties of this material as cold rolled:

MECHANICAL PROPERTIES OF COLD-ROLLED NO. 18 GAGE DURALUMIN SHEET.

Average thickness, in		0.0403
Proportional limit, lb. per sq. in		40 650
Elastic limit, lb. per sq. in		41 220
Yield point, lb. per sq. in		46 260
Tensile strength, lb. per sq. in		51 740
Modulus of elasticity, lb. per sq. in		9 757 000
Reduction of area, per cent		12.9
Elongation in 4 in., per cent		2.7
Elastic ratio		0.80
Scleroscope hardness number	Universal hammer	22.9
	Magnifier hammer	45.9
Brinell hardness number (15 kg., 1 mm., 30 sec.)		109.0
Indentation (cupping) values	For ⅞-in. ball, pressure at rupture, lb	420
	Depth of cup, in	0.085
	For ½-in. ball, pressure at rupture, lb	430
	Depth of cup, in	0.072

The sheets as rolled were first sheared into suitable sizes for the indentation (cupping) tests and into strips for subsequent machining into tension test specimens. The tension specimens were made sufficiently long so that a section for Brinell and scleroscope hardness tests could be cut from one end and a section for microscopic examination cut from the other end. The samples were all prepared in final form for testing prior to heat treatment. The tension test specimens were machined to the form shown in Fig. 1, first by milling the center section to about 0.505 in. wide and then to final form by draw filing 0.002 in. under this width at the center of the section. The center section was made slightly narrower than the width of the main gage length in order to ensure rupture at the center of the bar. Specimens for the indentation (cupping) tests were $2\frac{1}{2}$ by 4 in. in size for use with the large ball ($\frac{7}{8}$-in. diameter) and $1\frac{1}{2}$ by 4 in. for use with the small ball ($\frac{1}{2}$-in. diameter).

NATURE OF THE EXPERIMENTAL HEAT-TREATMENT WORK

The various sheet samples for test were heat treated in lots in a wire-wound electric-resistance furnace, maintaining temperature control to ±2° C. In accelerated aging tests of quenched samples a Freas electric oven, maintaining good control of temperature (±2° C.) up to 250° C., was employed. Drawing at 300° C. was done in an electric-resistance furnace. For each particular test, tension specimens and pieces for the cupping test were heat treated in triplicate, and sufficient material held in reserve to make duplication of any tests desired. In the case of air cooling after heating, the samples were withdrawn from the furnace and allowed to stand on a concrete floor. The time required for cooling to the ordinary temperature was 2 to 5 minutes, depending on the thickness of the sheet. In the case of furnace cooling, after the required time period of heating, the power was shut off, and the samples allowed to cool in the furnace over night. The time required for furnace cooling to the ordinary temperature was some 12 to 18 hours, depending upon the original temperature. In water quenching, the water bath was maintained at 20° C. ±1° C. In oil quenching, the oil bath (Houghton's No. 2 soluble quenching oil) was maintained at the ordinary temperature. In the case of samples aged in air at the ordinary temperature, the pieces were simply allowed to stand in the air on a shelf away from any special

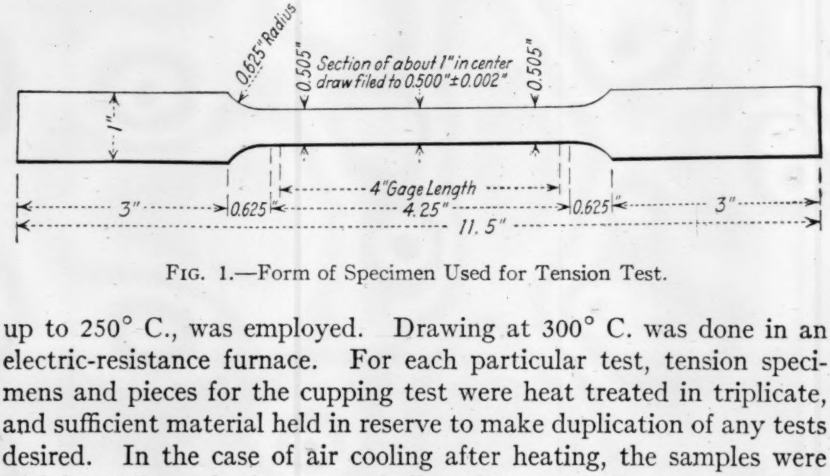

FIG. 1.—Form of Specimen Used for Tension Test.

heating or cooling effects. The average room temperature during the course of the aging tests was 20.3° C., with a maximum of 22.1° C., and a minimum of 14.6° C.

A series of exploratory heat treatments and tests was first carried out for the purpose of checking up certain prior work, notably that of Grard[1] and of Merica, Waltenberg, and Scott,[2] and also for determining the most suitable directions for carrying out the main bulk of the investigation. After these tests were completed, a schedule of heat treatment work was drawn up.

Annealing Heat Treatment.—Three main sets of annealing heat-treatment experiments were carried out, as follows:

1. Sets of the six gages of sheet were heated for 30 minutes at 300, 350, 400, 450, 500 and 550° C., and cooled in air. In this series, the effect of the temperature of heating for a constant time followed by air cooling was examined.
2. Sets of the six gages were heated for 30 minutes at the same temperatures as above, and cooled in the furnace. In this series, the effect of the temperature of heating for a constant time followed by furnace cooling was examined.
3. A set of the No. 18 gage sheet was heated for 1, 5, 10, 20, 30, 60, 120, 180, 300, and 600 minutes at 300, 350, 400, 450, 500 and 550° C., and cooled in air. In this series, the effect of the time period of heating at various temperatures followed by air cooling was examined.

After the annealing heat treatments described, the samples were aged in air at the ordinary temperature for six days prior to testing.

It is, of course, understood that duralumin will harden on aging when cooled in air from around 375° C., and in fact, when air cooled from a temperature even as low as 300° C., duralumin is in a slightly hardened condition, that is, harder and stronger than if cooled in the furnace from the latter temperature.

Quenching Heat Treatment.—Duralumin is normally used in the quenched-and-aged condition, and the quenching heat treatments carried out were more extensive and diversified than the annealing heat treatments above described. The various quenching heat treatments were as follows:

1. Sets of the six gages were heated for 30 minutes at 375, 400, 425, 450, 475, 500, 512, 525 and 550° C., quenched from these temperatures in water at 20° C., and then air aged for six days at the ordinary temperature prior to testing. Quenching below 375° C. has little effect. In this series, the effect of the temperature of quenching followed by air aging was examined.
2. A set of the No. 18 gage sheet was heated for 30 minutes at 512° C. (the preferred quenching temperature for the low-copper alloy), quenched in

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[1] C. Grard, *loc. cit.*

[2] P. D. Merica, R. G. Waltenberg, and H. Scott, "Heat Treatment of Duralumin," U. S. Bureau of Standards *Scientific Paper No. 347,* November 15, 1919.

water at 20° C., and then aged in air at the ordinary temperature for 0, 1, 2, 3, 5, 10, 24, 48, 72, 120, 240, 720 and 1440 hours prior to testing. In this series, the effect of the time period of aging at the ordinary temperature after quenching (from a preferred temperature) was examined.

3. A set of the No. 18 gage sheet was heated for 30 minutes at 512° C., quenched in water at 20° C., and then immediately aged for 1 hour at 20, 40, 60, 80, 100, 150, 200, 250 and 300° C., prior to testing. In this series, the effect of the temperature of aging, for a constant time, after quenching was examined.

4. A set of the No. 18 gage sheet was heated for 30 minutes at 512° C., quenched in water at 20° C., and then immediately aged at 100° C. for 1, 5, 10, 20, 30, 60, 120, 180, 300, and 600 minutes prior to testing. In this series, the effect of the time period of aging at a constant elevated temperature after quenching was examined.

5. A set of the No. 18 gage sheet was heated for 1, 5, 10, 20, 30, 60, 120, 180, 300, and 600 minutes at 512° C., then quenched in water at 20° C., and then air aged at the ordinary temperature for six days prior to testing. In this series, the effect of the time period of soaking at the quenching temperature was examined.

6. Sets of the six gages were heated for 30 minutes at 512° C., and then quenched in the following media: still air, an air blast, water, oil, iced brine, and boiling water. After quenching, the samples were air aged for six days prior to testing.

RESULTS OF THE MECHANICAL TESTS

Hardness tests were made on all the samples with the Shore scleroscope, using both the universal and magnifier hammers. In order to avoid the anvil effect so pronounced when testing light-gage sheet, samples of the various gages were backed with a piece of heavy gage sheet having had the same heat treatment as the sample tested. The mean of 10 determinations was taken as the hardness number. Hardness tests were also made on all the samples using a special small-ball Brinell machine, similar to that described by Goodale and Banks.[1] The load was 15 kg. and the diameter of ball 1 mm.; the time of imposed load was 30 seconds. The mean of three impressions was taken as the hardness number. The anvil effect was avoided by suitable backing as in the case of the scleroscope tests. The diameters of the ball impressions were read with a precision micrometer microscope.

Indentation (cupping) tests were made on all the samples to determine the drawing qualities of the sheet as affected by heat treatment. The drawability of duralumin sheet as affected by heat treatment has not been studied fully hitherto, the only reported work being that of Grard[1] and of Portevin and Le Chatelier,[2] the data of the latter applying to light aluminum-copper and aluminum-copper-manganese alloys, rather than to ordinary duralumin. The Olsen cupping machine was used for the tests, two sizes of balls being

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[1] S. L. Goodale and R. M. Banks, "Development of Brinell Hardness Tests on Thin Brass Sheet, *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, pp. 757–781 (1919).

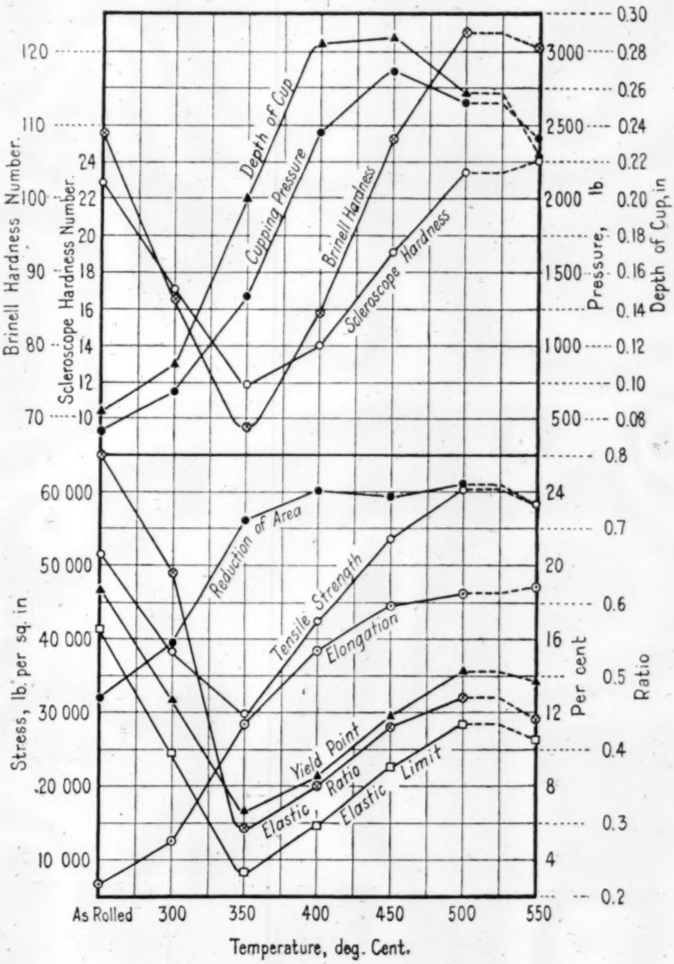

FIG. 2.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 30 minutes in the Range 300 to 550° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

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[1] C. Grard, *loc. cit.*

[2] A. Portevin and F. Le Chatelier, "Le Traitement Thermique des Alliages Légers d'Aluminiumà, Base de Cuivre," *Revue de Métallurgie*, Vol. 21, pp. 233–245 (1924); see also *Transactions*, Am. Soc. Steel Treating, Vol. 5, pp. 457–478 (1924).

employed, namely $\frac{7}{8}$-in. and $\frac{1}{2}$-in. diameter. The cupping tests were made on triplicate samples, and in duplicate on each piece; hence,

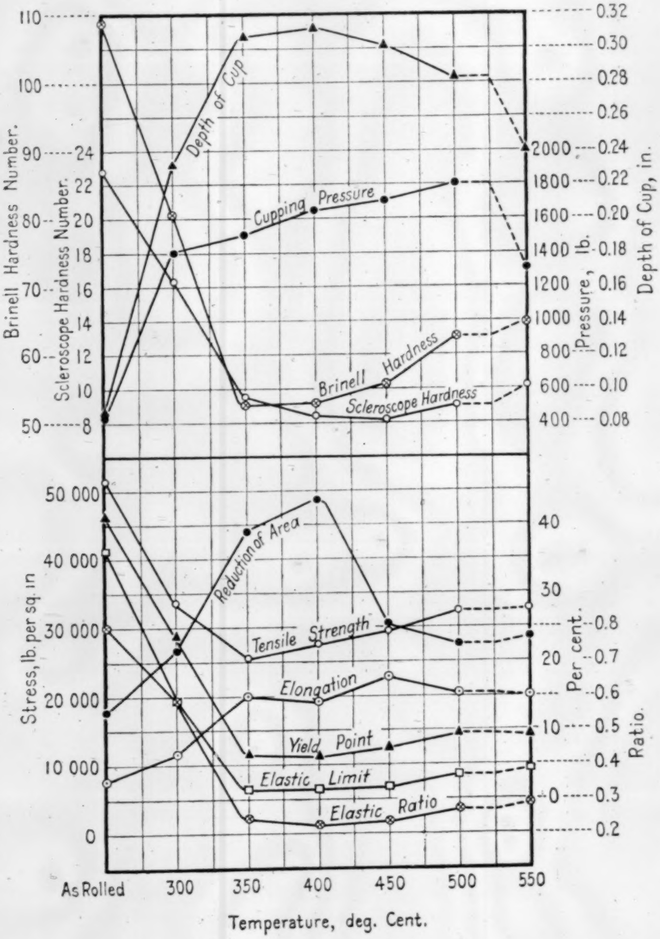

FIG. 3.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 30 minutes in the Range 300 to 550° C., Followed by Furnace Cooling; Aged 6 days at the Ordinary Temperature.

each value represents the mean of six determinations. The pressure in pounds at rupture and the depth of cup were determined.

Tension tests were made on all the heat-treated specimens in triplicate, using a 4000-lb. wire machine. A special light extensometer, with Ames dial gage reading to 0.0001 in., was used in taking data for the stress-strain diagrams. In these tests, the following data were taken: (1) proof stress (10 seconds load), for two extensions, that is, 0.10 and 0.15 per cent; (2) elastic limit, obtained by Johnson's method from the stress-strain diagrams; (3) yield point, for two extensions,

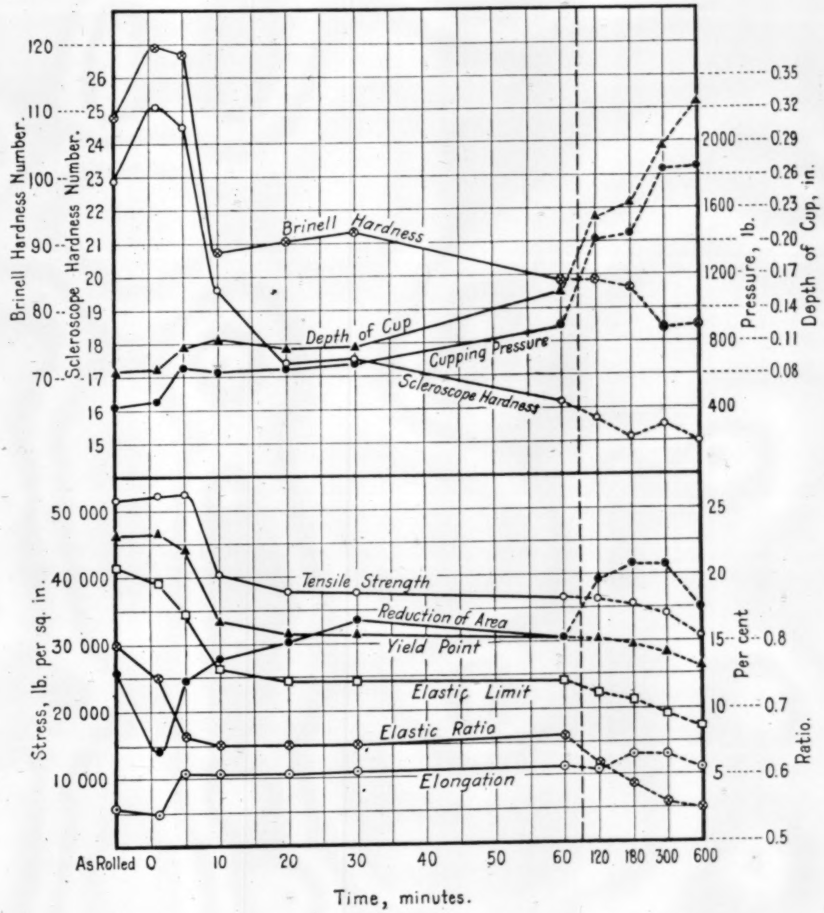

FIG. 4.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 300° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

that is, 0.01 and 0.02 in. on the gage length; (4) tensile strength; (5) reduction of area; and (6) elongation. Calculation was made for the elastic ratio, and the modulus of elasticity was calculated for typical samples. The actual proportional limits were obtained also from the stress-strain diagrams. In general, the methods of tension testing most lately recommended by the Society's Committee E-1 on Methods of Testing were followed.

All told, about 1000 tension test specimens were broken, using

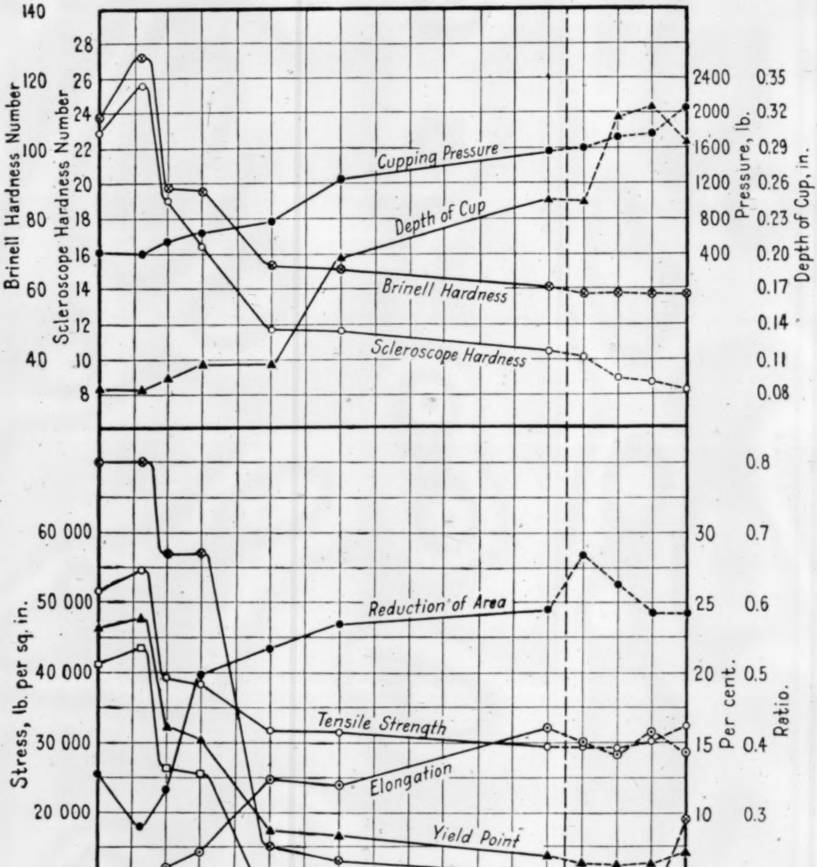

FIG. 5.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 350° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

the extensometer, and a correspondingly large number of tests made for scleroscope and Brinell hardness and cupping values. The summarized data obtained comprised 73 tables of numerical results.

Owing to the confines of space, the tabular data cannot be given in the present paper, and the results for five of the gages (Nos. 10, 14, 22, 26, and 30) are omitted entirely, except that the general nature of the results as regards these gages is discussed briefly. The data for the No. 18 gage sheet (on which the more comprehensive series of tests was made) are summarized in the graphs of Figs. 2 to 14, inclu-

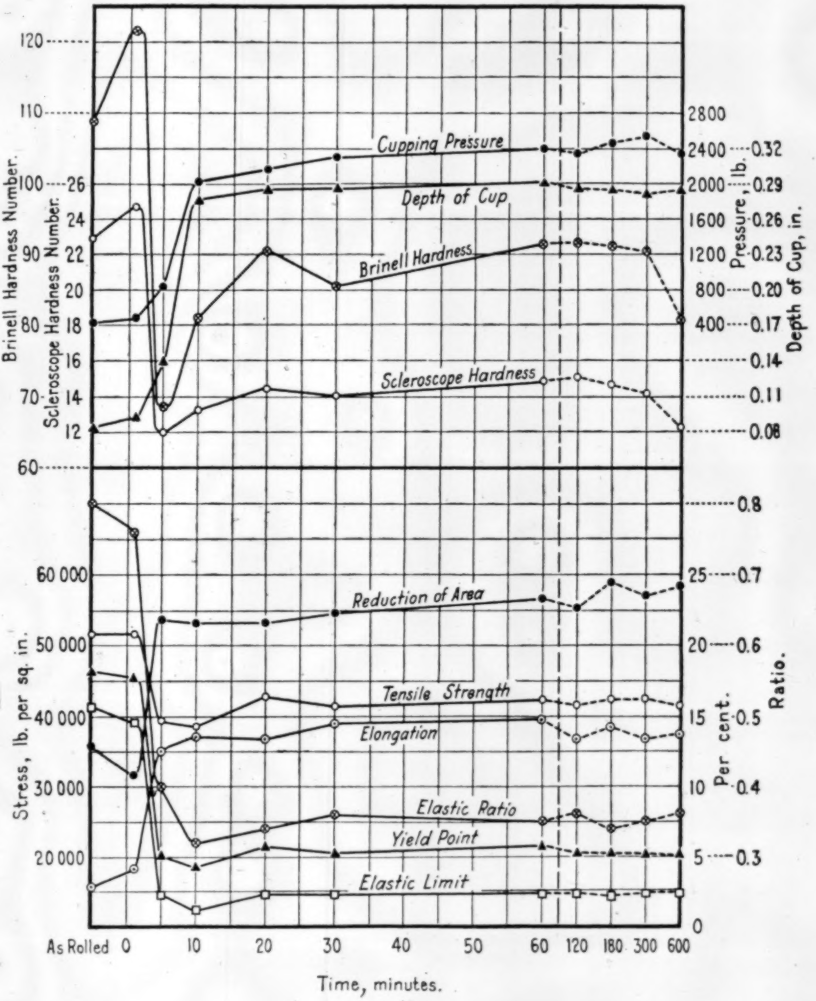

FIG. 6.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 400° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

sive. In these graphs, the original properties of the sheets, as cold rolled, are plotted for ready comparison. The graphs give the tensile properties, Brinell hardness, scleroscope hardness (universal hammer) and cupping values ($\frac{7}{8}$-in. diameter ball). The yield points as plotted

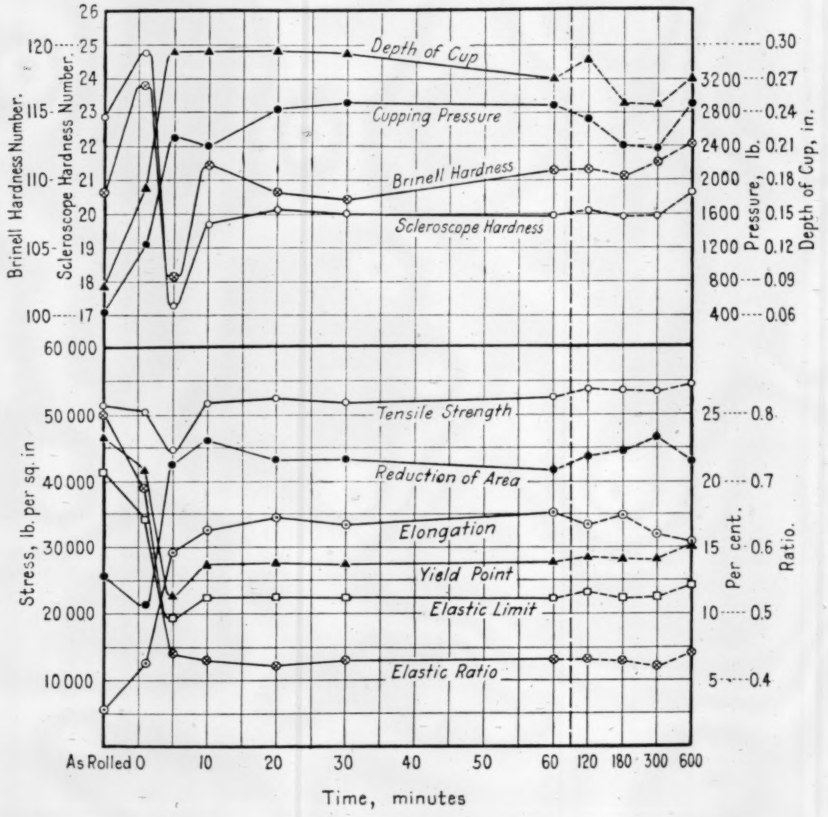

FIG. 7.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 450° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

are for extensions of 0.02 in. The data are self-explanatory, and the principal results of the tests are discussed briefly in later paragraphs.

In addition to the series of annealing, quenching, and aging heat treatments already described, sets of strips of the various sheet gages were heated in a furnace in such a way that the temperature at one end of the strips was 512° C. and at the other end 100° C. The strips were $11\frac{1}{2}$ in. long and $\frac{1}{2}$ in. wide. The time period of heating was

30 minutes, and then the strips were quenched in water at 20° C., followed by aging for six days at the ordinary temperature. Brinell and scleroscope hardness tests were made at ½-in. intervals along the lengths of these strips. The data for the No. 18 gage set are plotted in Fig. 15, and are of interest in showing how the resultant hardness. attained on aging, varies with the quenching temperature. This test has also been carried out by Merica, Waltenberg, and Scott[1], with results comparable to those obtained by the writer. When a strip is heated in the manner indicated, there is a temperature gradient along the length. At the hottest end of the strip, hardening ensues owing

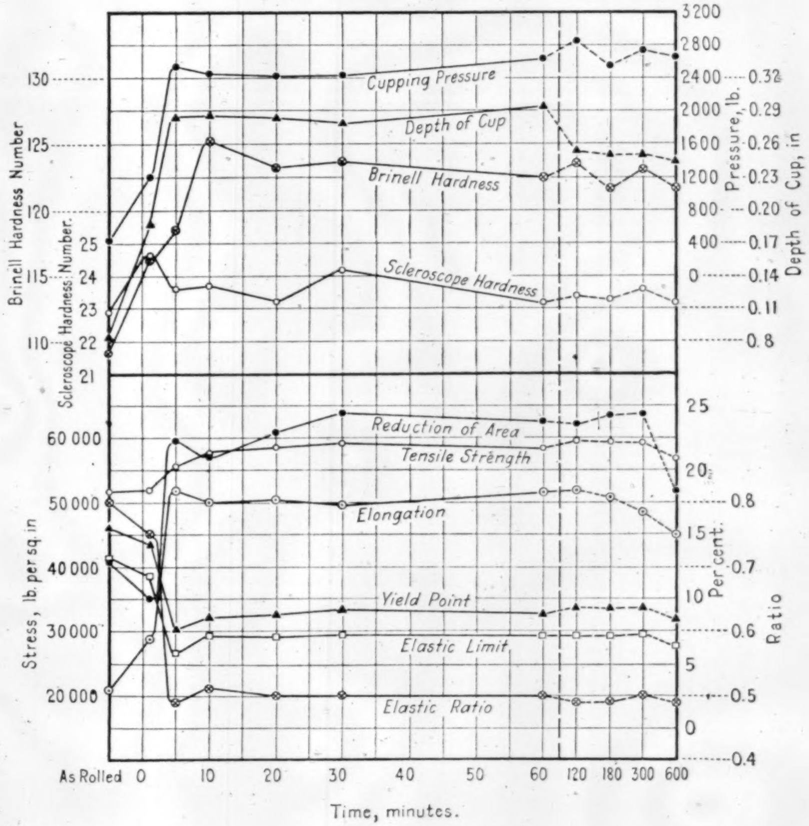

FIG. 8.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 500° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

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[1] P. D. Merica, R. G. Waltenberg, and H. Scott, *loc. cit.*

to the quenching and aging effects, while the center portion is soft due to the annealing effect.

DISCUSSION OF THE MECHANICAL TEST RESULTS

While the mass of data taken in connection with the tests on the various gages warrant detailed discussion, the confines of space permit only a brief résumé of the main results.

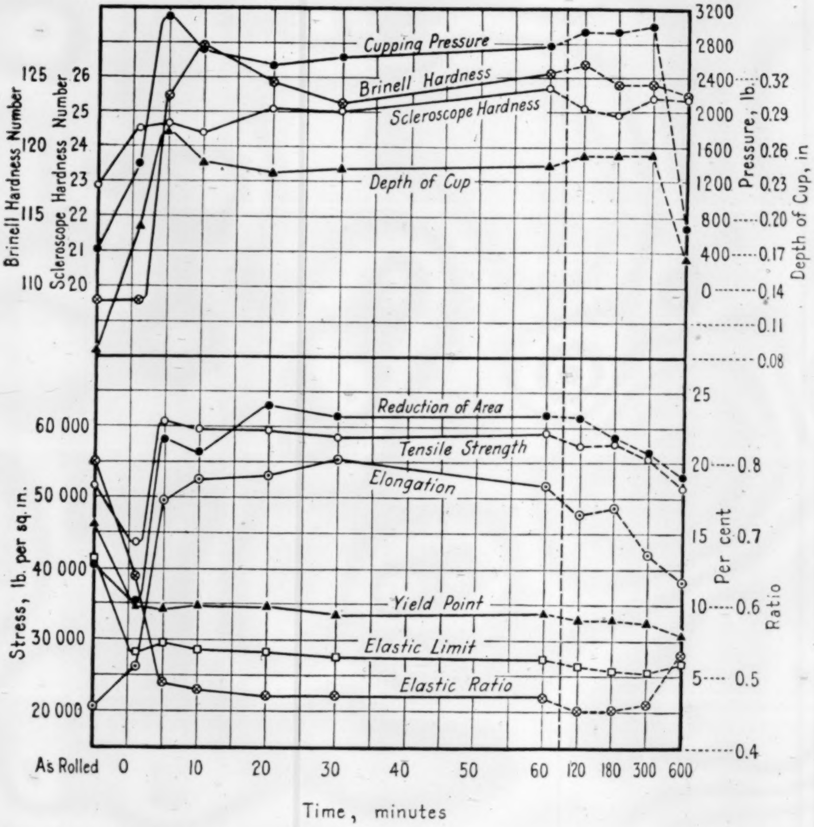

FIG. 9.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Heating for 1 to 600 minutes at 550° C., Followed by Air Cooling; Aged 6 days at the Ordinary Temperature.

Relations Among the Mechanical Properties.—The general trend of the relations among the mechanical properties of duralumin is indicated in the graphs of Figs. 2 to 14, inclusive. The values for the elastic limit, yield point, tensile strength and elastic ratio parallel one another, irrespective of the treatment, that is, high tensile strength is accompanied by high values for the elastic limit, yield point, and elastic ratio. The general tendency is for the reduction of area to decrease with increase in strength and related tensile properties. There are certain exceptions to this general statement, but these are owing to specific heat treatments which would be expected to give the results obtained. The elongation tends to decrease with increase in strength, and *vice versa*. The Brinell hardness most closely parallels the tensile strength, and in most cases small change in strength is reflected in measurable change in Brinell hardness. The small-ball Brinell-hardness test on sheet is a much more sensitive

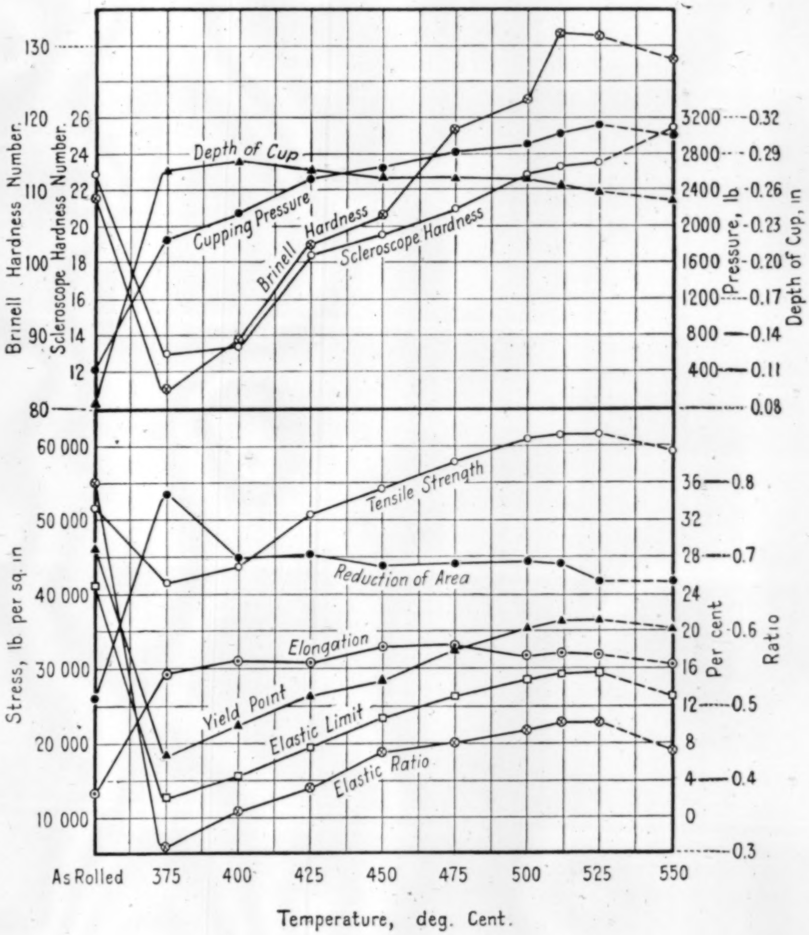

FIG. 10.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Water Quenching in the Range 375 to 550° C.; Aged 6 days at the Ordinary Temperature.

indicator of both hardness and strength changes than is the scleroscope test. The greater the strength and related properties the greater the hardness. The scleroscope-hardness values more closely parallel the yield-point figures than do the Brinell-hardness values, and while the scleroscope is not a sensitive indicator of either strength or hardness changes in the case of sheet, it apparently reflects yield-point changes. While the Brinell-hardness and scleroscope-hardness curves in Figs. 2 to 14, inclusive, do show considerable parallelism, it seems very doubtful that there is any definite relation between these properties in the case of duralumin. Calculation of numerous

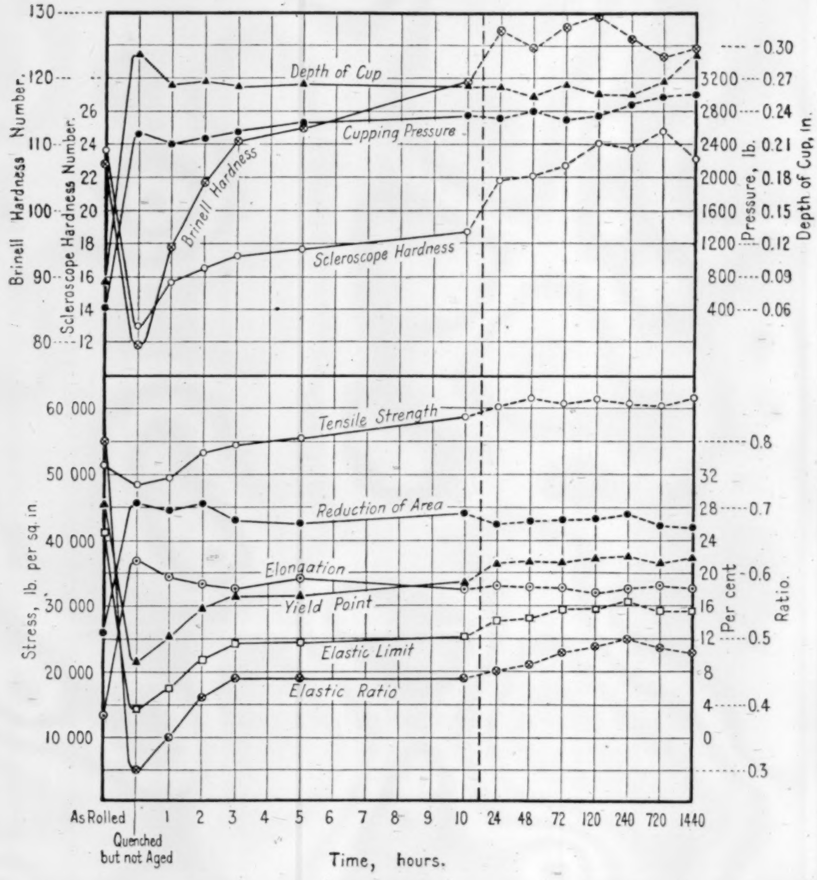

FIG. 11.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Water Quenching from 512° C., Followed by Aging for 1 to 1440 hours at the Ordinary Temperature.

relative values for the two properties shows that the numerical relation of the Brinell hardness to the scleroscope hardness (regular hammer) varies between 4.2 and 6.8. Roughly, it may be said that the

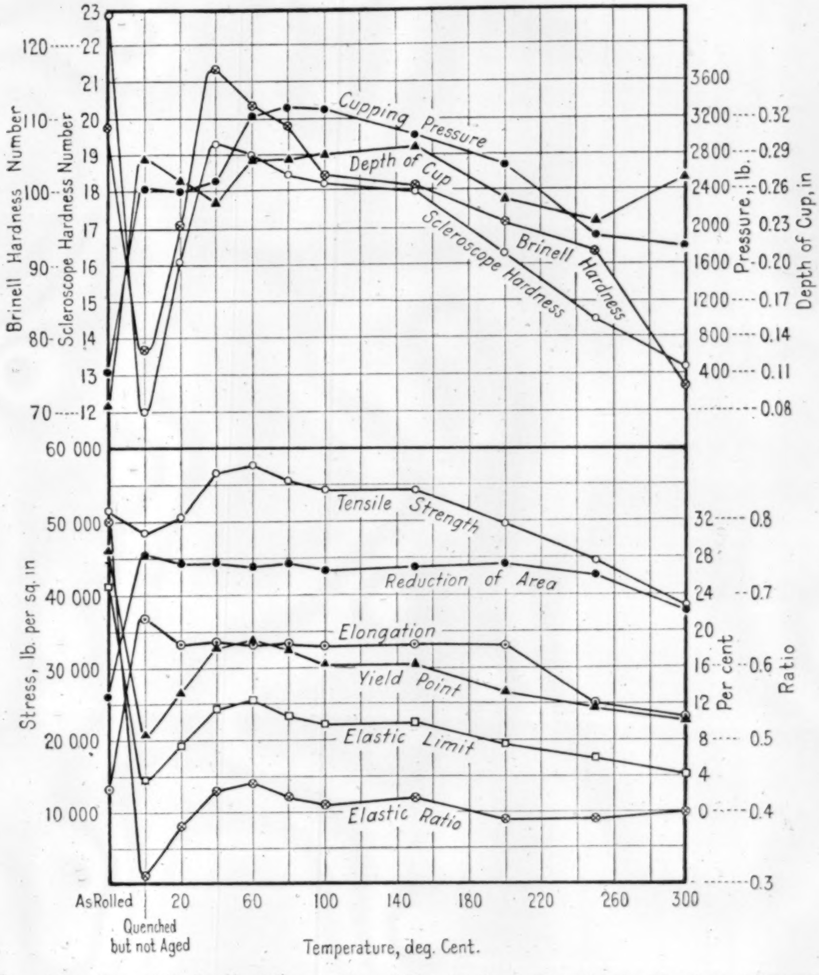

FIG. 12.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Water Quenching from 512° C., Followed by Aging for 1 hour in the Range 20 to 300° C.

Brinell hardness (15 kg., 1-mm. ball) is six times the scleroscope hardness (regular hammer).

The ductility of duralumin sheet, as measured by the depth of cup, is a function of the reduction of area, and, taken generally, the greater the reduction of area the greater the depth of cup. The depth

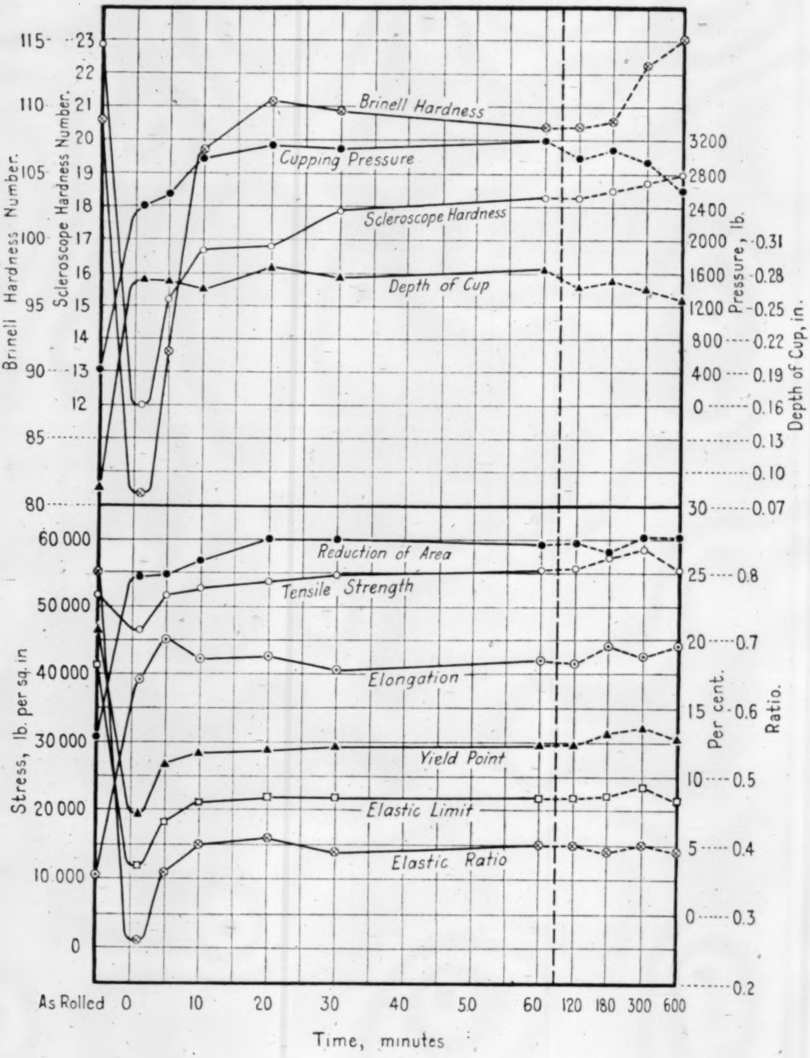

FIG. 13.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Water Quenching from 512° C., Followed by Aging for 1 to 600 minutes at 100° C.

of cup is also a function of the tensile strength, and, other things being equal, the stronger a sheet, the greater its indentation in the cupping test. The pressure required to rupture sheet in the cupping test is related both to the strength and hardness and to the depth of cup; in the main, the harder and stronger a sheet is, the greater the pressure required to rupture. This applies especially to quenched-and-aged sheet. Cold-worked sheet is weak in cupping pressure, and in dead soft annealed sheet the pressure seems more closely related to the depth of cup than to any other property.

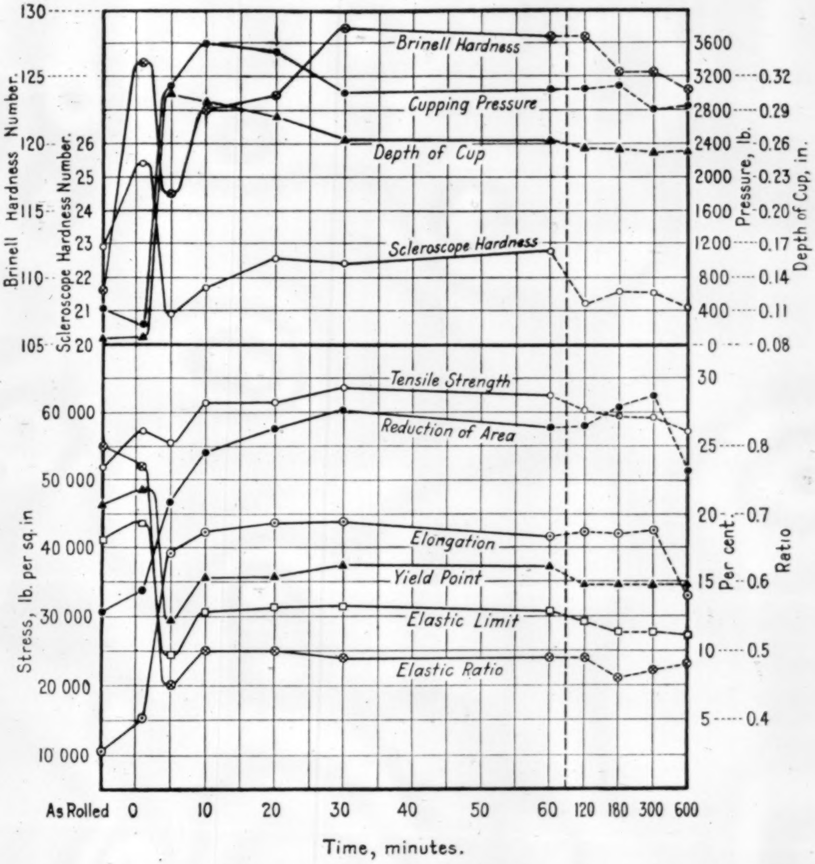

FIG. 14.—Some Mechanical Properties of No. 18 Gage Duralumin Sheet as Affected by Soaking for 1 to 600 minutes at 512° C., prior to Water Quenching at 512° C., Followed by Aging 6 days at the Ordinary Temperature.

Elastic Limit and Stress-Strain Diagrams.—As will be recalled, the tension tests in this investigation were made with an extensometer

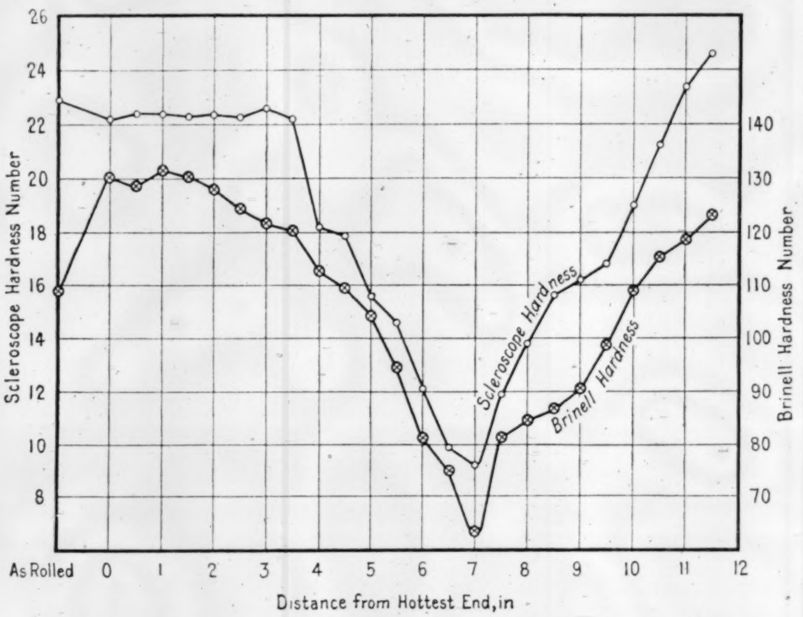

FIG. 15.—Hardness of No. 18 Gage Duralumin Sheet Strip as Affected by Water Quenching, with One End at 512° C. and the Other End at 100° C.; Aged 6 days at the Ordinary Temperature.

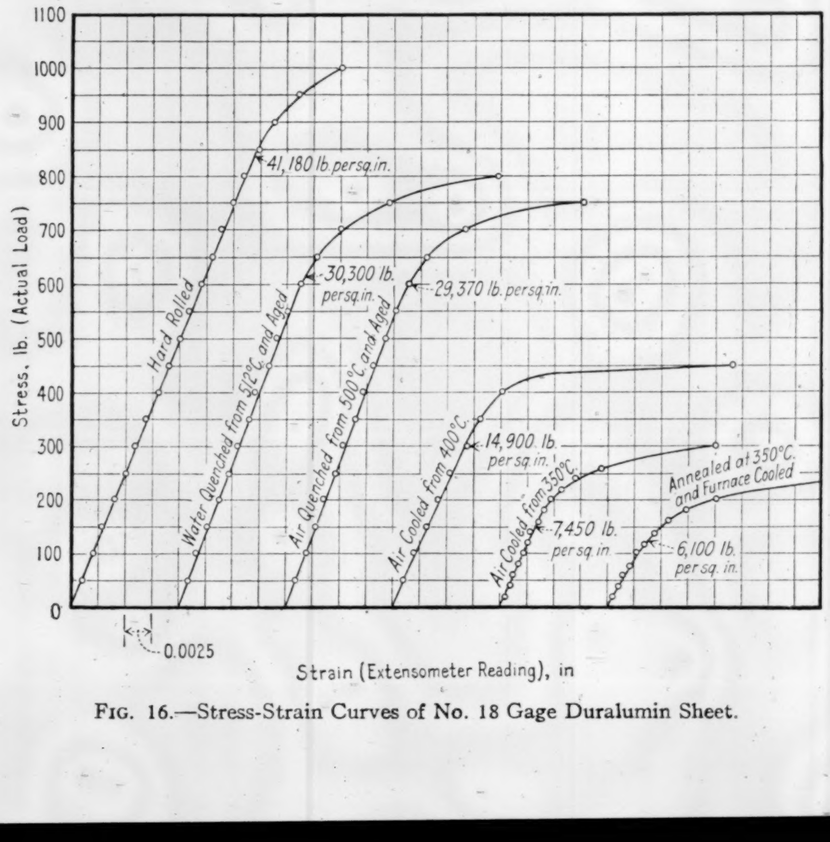

FIG. 16.—Stress-Strain Curves of No. 18 Gage Duralumin Sheet.

for determining the elastic limit, and the elastic limit was obtained by Johnson's method. The elastic limit as obtained by this method is higher than the proportional limit, and the divergency between these two values depends upon the heat treatment given to the material. Fig. 16 shows the type of stress-strain curves obtained, those given being for No. 18 gage low-copper sheet as hard rolled and after different treatments. The six curves shown in the figure are all plotted to the same scale, taking a new point of origin for each successive curve from left to right. The values indicated on the curves are the respective "Johnson" elastic limits. It will be seen that the rate of deflection of the graphs tends to fall off with increasing softness and decreasing strength of the sheet, that is, the straight-line parts of the graphs slope more on passing across Fig. 16 from left to right This is to be expected since the elastic modulus of the softer sheets is less. There is good parallelism among the first three graphs on the left-hand side of Fig. 16 indicating similar elastic moduli (see also Table I). The modulus of elasticity of duralumin is not greatly affected by heat treatment, other than by a softening anneal. Young's modulus for quenched-and-aged duralumin may be taken as around 10,500,000 lb. per sq. in.

TABLE I.—SUMMARY OF TENSILE PROPERTIES OF NO. 18-GAGE DURALUMIN SHEET AS AFFECTED BY VARIOUS HEAT TREATMENTS.

Treatment	Proportional Limit, lb. per sq. in.	Elastic Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Reduction of Area, per cent	Elongation in 4 in., per cent	Modulus of Elasticity, lb. per sq. in.
As rolled (not treated)	40 650	41 220	51 740	12.9	2.7	9 757 000
Air cooled from 350° C.[a]	7 210	8 800	29 900	22.5	11.3	7 648 000
Furnace cooled from 350° C.[a]	5 080	6 100	25 310	39.0	15.0	5 540 000
Air cooled from 400° C.[a]	14 080	14 880	42 250	24.1	15.4	9 464 000
Air cooled from 500° C.[a]	27 770	28 500	60 480	24.4	18.5	10 210 000
Furnace cooled from 500° C.[a]	7 430	8 800	32 290	22.6	15.3	5 444 000
Water quenched at 512° C.[a]	28 470	29 130	61 170	27.3	17.6	10 545 000
Iced-brine quenched at 512° C.[a]	28 620	29 170	61 030	26.7	19.6	10 450 000
Oil quenched at 512° C.[a]	28 570	29 270	61 670	26.3	18.6	10 540 000
Freshly quenched in water at 512° C.[b]	14 080	14 850	48 280	28.3	21.5	8 700 000
Water quenched at 512° C.; drawn 1 hour at 100° C	20 740	21 830	55 250	27.1	18.5	10 293 000
Water quenched at 512° C.; drawn 10 hours at 100° C	20 440	21 430	55 590	27.7	19.8	9 635 000
Water quenched at 512° C.; drawn 1 hour at 200° C	18 930	19 610	49 900	27.3	18.3	9 400 000
Water quenched at 512° C.; drawn 1 hour at 300° C	14 710	15 200	38 430	22.1	10.3	9 252 000
Water quenched at 512° C.; aged 1440 hours in air	28 290	29 130	61 650	25.7	18.3	10 582 000

[a] After heating for 30 minutes at the temperature indicated, and aged 6 days at the ordinary temperature after the treatment.

[b] Not aged.

Lüders Lines and Slippage.—In the tension testing of duralumin, after the elastic limit has been passed and more particularly above the yield point and near the breaking load, increased loading is accompanied by a series of slips, the stress apparently increasing in an oscillatory manner. The slips are accompanied by a series of audible clicks or sounds. These sounds are high pitched in the case of thin sheet, not unlike the tinkle of Japanese glass chimes; with thick sheet, the sounds are low in note, being on the order of a grunt. The phenomenon has been described previously by Portevin and Le Chatelier,[1] who find that the slippage and audible clicking are most pronounced in freshly quenched samples and disappear after aging is completed. During the course of the slippage, the lines of slip known as Lüders, Hartmann, or Piobert lines appear on the bars. The phenomenon has been noticed in some other metals and alloys, for example, in certain aluminum-zinc alloys, in mild steel (tested between 80 and 250° C.) and in 25-per-cent nickel steel.

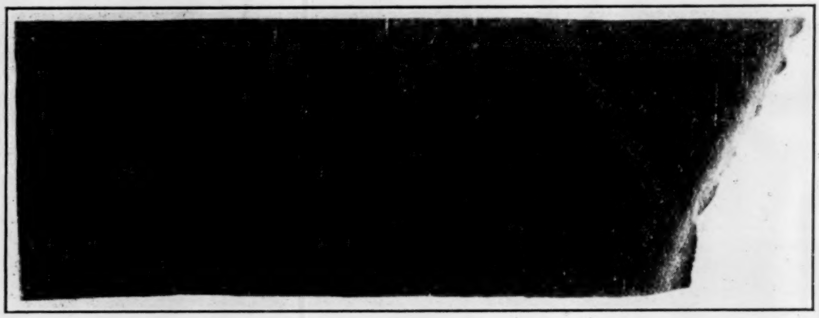

FIG. 17.—Lüders Lines in Tension Test Specimen of Duralumin (× about 2¾).

The phenomenon of slippage was studied by the author in the present investigation, and the audible clicking in tension testing was heard with virtually all the bars (about 1000), being noticeably absent mainly in those samples heated to various temperatures and furnace cooled. The author's observations as to slippage and clicking are different from those reported by Portevin and Le Chatelier, who find, for example, that the phenomena disappear in quenched duralumin after the aging is completed. A summary of the observations made by the author is as follows: Audible clicks accompanied by heavy slippage were heard in the cold-rolled sheets, except in the two lightest gages. They were heard in all bars heated, air cooled, and aged, except for bars heated more than 3 hours at 550° C. Clicking was absent in bars heated in the range 300 to 550° C. and furnace cooled, except in the case of the No. 26 and No. 30 gage sheet heated at 400 and 450° C., which gave soft faint clicks. Freshly quenched bars gave the loudest clicks for a given gage, and on accelerated aging after quenching the clicking starts at higher loads (nearer the breaking point) with increase in the time of aging. When bars are soaked for a long time prior to the quench (longer than 3 hours), no clicking occurs in tension testing. Bars heated at 550° C. prior to the quench do not give clicks.

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[1] A. Portevin and F. Le Chatelier, "Sur un Phénomené Observè Lors de l'Essai de Traction d'Alliages en Cours de Transformation," *Comptes Rendus*, Vol. 176, pp. 507–510 (1923); and *loc. cit.*

The clicks are the accompaniment of slippage, and this slippage gives rise to a series of serrated lines at about 30 deg. with the horizontal across the face of the bars. These lines may be properly regarded as Lüders lines, that is, geometrical lines caused by deformation. The actual fracture of a ruptured tension bar normally takes the same angle as the Lüders lines in duralumin, but in the opposite direction to the bulk of the lines, as shown in Fig. 17. In some cases, the lines run mainly in one direction, while in others there are two series of lines crossing each other, both at about 60 deg. with the direction of the stress. In some bars, reversal of direction of the lines takes place about $\frac{1}{4}$ in. from the break. According to Portevin and Le Chatelier, the phenomenon of slippage and clicking is connected with the transformation in duralumin on aging which causes hardening and strengthening. This apparently is true since it is most pronounced in quenched duralumin and noticeably absent in furnace-cooled samples. Observations by the author of fractures of numerous tension test specimens of duralumin show that the bars tend to break at an angle of 60 deg. with the direction of the stress and conjugate to the bulk of the Lüders lines. Evidently, the first Lüders line which appears causes local constriction and determines the position of rupture.

CONCLUSIONS FROM THE MECHANICAL TESTS

Table I gives a summary of tension test data for the No. 18 gage sheet after various heat treatments, which will serve to indicate the order of results obtained. The main conclusions reached from a study of the mechanical-test results are summarized as follows:

1. On heating cold-worked duralumin sheet in the range 300 to 550° C. for 30 minutes, followed by air cooling and aging, the elastic ratio, elastic limit, yield point, tensile strength and hardness drop to minimum values for the temperature 350° C. and then rise to maxima for 500° C. The temperature 350° C. is a suitable annealing temperature for duralumin when followed by air cooling. The values rise on air cooling from above 350° C. since the alloy hardens (on aging) following air quenching, although the strength and hardness attained on air quenching are never so high as those secured by water quenching. The elongation tends to increase with the temperature from

which cooled, but depends upon the thickness of the sheet. The reduction of area tends to be a maximum corresponding to greatest softening, and the depth of cup tends to parallel the reduction of area. The tensile strength of sheet air cooled from 350° C. is about 30,000 lb. per sq. in., as compared with 60,000 lb. per sq. in. when air cooled from 500° C. (fully air aged in both cases), while the corresponding Brinell-hardness numbers are about 70 and 125.

2. On heating cold-worked duralumin sheet in the range 300 to 550° C. for 30 minutes, followed by furnace cooling and air aging, the elastic ratio, elastic limit, yield point, tensile strength and hardness attain minimum values for the temperature 350° C., and then rise slowly with increasing temperature from which cooled. Furnace-cooled sheet is always softer and weaker than air-cooled sheet, both being cooled from the same temperature and fully air aged. Furnace cooling gives very high values for the reduction of area, the maxima coming at 400° C. and running as high as 45 per cent, as against 25 per cent for air cooling. The depth of cup is also very high for furnace-cooled sheet. Furnace cooling causes a great drop in the elastic limit as compared with air cooling. The tensile strength of sheet furnace cooled from 350° C. and fully air aged is around 25,000 lb. per sq. in., as against 33,000 lb. per sq. in. when furnace cooled from 500° C., and the corresponding Brinell-hardness values are about 53 and 63.

3. The effect of the time period of heating in the range 300 to 550° C. followed by air cooling and aging is rather complex. The data show that the first effect of heating, that is, as indicated by short-time heatings, is a hardening and strengthening; a rise of over 3000 lb. per sq. in. in the tensile strength and 15 points in Brinell hardness has been noted, for example, on heating cold-rolled sheet for 1 minute at 350° C. This phenomenon has been observed many times with other metals and alloys. Increase in the time period of heating at 300° C. causes a gradual drop in the elastic ratio, elastic limit, yield point, tensile strength and hardness, with corresponding rise in the reduction of area, elongation, pressure at rupture and depth of cup. On heating at the higher temperatures, the properties decrease or increase as just indicated up to a certain time period of exposure. With longer exposures, a reverse effect takes place. This behavior is apparently connected with certain solubility conditions in the alloy. Thus, on heating at 350° C., the tensile strength and related properties decrease with increasing time period of exposure up to 180 minutes and then rise with longer heating periods. The long soaking at this low temperature may result in an excess of $CuAl_2$ solution, and on air quenching and aging the greater amount of

$CuAl_2$ in solution (than would be in solution with shorter heating periods) causes greater hardening since a greater amount would precipitate out. In general, for a given time period of heating, the higher the temperature of heating the stronger and harder is the alloy. This applies to heating at 400° C. and above, short of burning. On heating below 400° C., the higher the temperature of heating the greater the softening and loss of strength, except for reverse effects caused by long-time heatings, for example, at 350° C. The exact mechanism of the change in properties of duralumin, as determined by any given heat treatment in relation to some other treatment, must be considered for individual treatments, and it is not possible to draw sweeping generalities. On heating cold-worked duralumin, which is subsequently air cooled and then aged, there are at least two effects taking place which may mask or offset one another. One effect is the normal softening which would be expected on annealing any cold-worked alloy. The other is the hardening-on-aging effect which always tends to take place in any alloy in which the solid-solubility curve of one component in the base slopes with rise in temperature, indicating increase in concentration.

4. When duralumin is water quenched and then air aged at the ordinary temperature, the tensile strength and hardness increase with increase in the quenching temperature short of actual melting of the $CuAl_2$-aluminum eutectic. The elongation and reduction of area tend to fall off with increasing quenching temperature, as does the depth of cup, while the cupping pressure increases. Duralumin water quenched at 512° C. and fully aged at the ordinary temperature may have an elastic ratio of 0.48 to 0.50, elastic limit of 29,000 to 33,000 lb. per sq. in., yield point of 36,000 to 39,000 lb. per sq. in., tensile strength of 61,000 to 68,000 lb. per sq. in., reduction of area of 22 to 26 per cent, elongation of 14.5 to 17.5 per cent, and Brinell hardness of 130 to 145. The indentation values vary considerably depending upon the thickness of sheet tested.

5. When duralumin is water quenched at 20° C. from suitable temperature, such as 512° C., and then aged in air at the ordinary temperature, the strength and hardness rise rapidly at first and then more gradually, until the practical maximum enhancement of properties is attained in 72 hours, as shown by the present tests. Various statements are made in the literature to the effect that full aging requires four to ten days. The data obtained in the present investigation show, for the alloys used, that the tensile strength and related properties rise progressively with lapse of time on maturing at the ordinary temperature up to 72 hours and that further aging has

little effect. The strength attains its maximum in 24 hours, but the elastic limit lags behind and does not become maximum until 72 hours. The reduction of area and elongation tend to decrease during the earlier stages of the aging but become substantially constant in 2 hours. The hardness parallels the elastic limit, becoming maximum in 72 hours.

6. When quenched duralumin is aged for a constant time (1 hour) at progressively increasing temperatures up to 300° C., the tensile strength and related properties rise progressively with increase in the aging temperature up to 60° C., and then fall off for higher temperatures. Roughly, the strength increases for aging temperatures up to 60° C., then drops off a little and remains substantially constant for temperatures up to 150° C., and then drops markedly. The maximum values attained on accelerated aging for any of the times and temperatures examined are always lower than the values secured on aging at the ordinary temperature following quenching. These results are as would be expected, although contrary to statements made in the literature to the effect that accelerated aging is simply a speeding-up process of ordinary air aging.

7. On varying the time period of aging over a considerable range (1 to 600 minutes) at 100° C. in the case of freshly quenched duralumin, it is found that the tensile strength and related properties increase with increase in the aging time up to 5 hours and then decrease. The maximum strength attained in all the accelerated aging tests was around 58,000 lb. per sq. in., with Brinell hardness of about 113; this may be compared with 61,000 lb. per sq. in. and 130 for sheet of the same gage quenched under the same conditions but air aged at the ordinary temperature.

8. The time period of soaking at the quenching temperature prior to water quenching and air aging is relatively unimportant. Soaking for 30 minutes is ample for sheet stock, but bars and forgings should be heated longer to ensure uniformity of temperature throughout the mass. Long periods of soaking are deleterious owing to gassing of the stock by the furnace atmosphere. Thus, heating for more than 2 hours prior to the quench gives a weakened alloy and lowered properties.

9. Roughly, the quenching medium has little effect upon the resultant mechanical properties. In general, the more drastic the quenching the greater the strength and hardness and the lower the elongation. The actual differences in mechanical properties as affected by different quenching media are small but still measurable. Of the quenching media used, oil and water give the best all around results and are generally most suitable.

10. The most preferable general heat treatment procedure for ordinary duralumin (for example, the low-copper alloy used in this investigation) carried out for the purpose of obtaining the greatest strength and hardness consists in quenching in water at 500 to 512° C., followed by aging for 3 or 4 days at the ordinary temperature.

11. While the foregoing conclusions apply generally to both the low-copper and high-copper alloys, and the effect of the additional 2 per cent copper in the latter is generally slight, it is found that the elastic limit of the high-copper alloy, particularly in annealed samples, is greater than that of the low-copper alloy. The additional copper gives a stiffening effect. Also, the highest tensile strengths in quenched and aged samples were obtained with the high-copper alloy.

DISCUSSION

Mr. Lessells.

MR. J. M. LESSELLS[1] (*presented in written form*).—The paper by Mr. R. J. Anderson gives some very interesting data on the mechanical properties of duralumin sheet and it forms a welcome contribution to the literature. The writer believes, however, there are one or two points which should be discussed.

In the first place the author appears to emphasize the elastic limit value of this material, although this value was presumably obtained by an extensometer which averaged the extensions on both sides of the test bar. It should be borne in mind that on such thin materials the question of alignment greatly influences the proper determination of the elastic limit and from our experience on larger sections we would strongly recommend a mirror type of extensometer which gives readings of extension for both sides of the test bar.

The large discrepancy between the proportional limit (called the elastic limit by the author) and the Johnson elastic limit as shown by some of the results in Table I is worthy of the attention of Committee E-1 on Methods of Testing. The writer has observed cases in steel where the proportional limit was 27,000 lb. per sq. in., whereas the Johnson elastic limit was 80,000 lb. per sq. in., showing therefore greater divergence than indicated in these duralumin tests.

The paragraph of the paper on Lüders lines and slippage is of special interest to the writer. If reference be made to Figs. 7, 8 and 9 of a paper by Lynch, Mochel and McVetty presented last year,[2] where autographic tension test diagrams of medium carbon steel and 5-per-cent nickel steel are shown, it will be noted that for the case of 200° C. the diagram instead of being a smooth line after the yield point has been passed consists of a series of vertical lines caused by slippage of the crystals during deformation. While these diagrams were being obtained distinct sounds emanating from the test bar could be heard and of course the pendulum of the Amsler machine continued to oscillate giving the serrated diagram as shown. No such phenomenon has been observed in tests at normal temperature but this has always been observed while testing steels at 200° C. That this is a characteristic of the material and not of the machine can be at once seen from the fact that the same serrated diagram of extension has now been obtained by Mr. McVetty during the loading for long-time tests of stainless iron using the special extensometer designed by him. Probably we have here some further support for the amorphous theory. **Mr. Lessells.**

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[1] Engineer in Charge, Mechanics Section, Research Laboratory, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] T. D. Lynch, N. L. Mochel and P. G. McVetty, "The Tensile Properties of Metals at High Temperatures," *Proceelings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 5 (1925).

In conclusion, the writer sees no mention of the work done by the Alloys Research Committee on Aluminum Alloys and published in their Eleventh Report. Since they discuss some of the papers mentioned by the author, attention is drawn to this fact so that no work will be duplicated.

Mr. Templin. MR. R. L. TEMPLIN.[1]—There are two points to which I should like to call attention. The first of these is the considerable difference in values for the modulus of elasticity as indicated in Table I, and the conclusions the author draws therefrom. From literally hundreds of tests on material of similar composition, we have been unable to obtain such low modulus values as indicated therein. We find that the modulus value is very nearly 10,000,000 lb. per sq. in., irrespective of the condition of the metal.

The second point has to do with the Brinell hardness values shown, which in general are higher than those obtained using the international standard combination of 500-kg. load on a 10-mm. ball. The use of a load out of proportion to this combination probably accounts for the high Brinell values obtained.

Mr. Anderson. MR. R. J. ANDERSON (*author's closure by letter*).—In reply to the remarks made by Mr. J. M. Lessells, the elastic limits plotted in the graphs shown in the paper were taken from stress-strain diagrams, using Johnson's method, and the figures so obtained are necessarily higher than the true elastic (proportional) limits. Mr. Lessells' statement regarding the necessity of alignment in testing thin specimens is quite to the point, and a mirror type of extensometer which gives readings of extension for both sides of the test specimen is preferable to the type of extensometer used by the author. The phenomenon of slippage in materials other than duralumin is well known, as was mentioned by the author in the present paper. This phenomenon is evidently connected with the condition of the material tested (at least this is so in the case of duralumin), and the author does not see any connection with the amorphous theory. Regarding the work described in the Eleventh Report of the Alloys Research Committee, this is well known, and the confines of space prevented citing all the previous work bearing on the present paper.

It is clearly stated in the paper that the modulus of elasticity of duralumin is not greatly affected by heat treatment, other than by a softening anneal. The numbers obtained in Brinell hardness tests naturally depend upon the load and diameter of ball.

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[1] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

EFFECTS OF SIZE AND SHAPE OF TEST SPECIMEN ON THE TENSILE PROPERTIES OF SHEET METALS

By R. L. Templin[1]

Synopsis

This investigation covers an extensive series of tension tests on three types of sheet metals, namely: (1) material having a comparatively high tensile strength and a low elongation, (2) material having a comparatively low tensile strength and a high elongation, and (3) material having both a high tensile strength and a high elongation; the purpose being to determine the effects of size and shape of test specimen upon the tensile properties.

The major portion of the work includes tests on five different thicknesses of each type of metal ranging from $\frac{1}{64}$ to $\frac{1}{4}$ in. thick. This portion of the work is concerned with the width and length of the reduced section of the test specimen, maintaining specimen end widths and radii of fillets with a definite relationship to the reduced widths of the test specimens.

The second portion of the investigation concerns the effects of radius of fillets and end width on the mechanical properties of the material used, maintaining the width and length of the reduced section of the specimens constant. This portion of the investigation was performed on the three types of materials indicated above but on one thickness only.

The results of the investigation indicate quite definitely that the tensile strength and yield point of the material are affected in only a few extreme cases by the size and shape of the test specimen used. The elongation on the other hand, is affected quite seriously by the total cross-sectional area of the test specimen rather than by the form of the test specimen, except in so far as the form of the test specimen causes fractures to occur outside of the middle third of the reduced section. Heretofore, the value of the ratio of width to thickness of the reduced section of a test specimen has been considered one of the major factors, if not the most important, affecting the elongation.

A few supplementary tests are included in the investigation for the purpose of verifying the relationship found to exist between total cross-sectional area and elongation and for the purpose of determining whether or not this relationship will hold in extreme cases of cross-sectional shapes of test specimens.

As a result of these tests and limitations imposed by preparation and machining, a size and form of satisfactory tension test specimen is indicated.

Introduction

As a part of the problem of defining the mechanical properties of aluminum and its alloys, instigated about seven years ago in the laboratories of the Aluminum Company of America, it became necessary to decide upon some size and shape of specimen, suitable for

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[1] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

use in tension tests of sheet metals. The sheet metals for which this specimen was to be used varied in thickness from 0.005 to 0.250 in. Appreciating the impracticability of selecting a single size of specimen for such material that would give directly comparable mechanical property values for all thicknesses within the range just indicated, it was decided to give much consideration to such factors as ease of preparation, duplication, handling and testing. A study of the sizes and forms of tension test specimens which had been used or were then in use, together with the factors just noted, led to the adoption of the specimen shown in Fig. 1. Many thousands of tension tests have been made of sheet metals using this specimen with very satisfactory results from a commercial viewpoint. These tests have

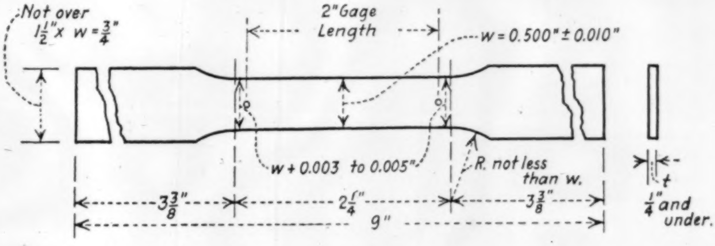


FIG. 1.—Tension Test Specimen.

included specimens not only of aluminum and its alloys in their various tempers and conditions, but also of magnesium, brass, copper, steel, nickel, zinc, bronze and other metals.

Other forms and sizes of tension specimens for sheet metals, however, have been used in other laboratories and by other investigators apparently with satisfactory results. When attempts are made to compare results obtained on the same kind and thickness of sheet metal using different sizes and forms of specimens certain differences are usually noted in the ductility values. Further attempts to compare dissimilar metals of different thicknesses using different test specimens often lead to much confusion. Recourse to the available literature on the subject helps somewhat but often not much, due to the fact that the equation developed to compare elongation values involves certain constants which at this time have not been determined with sufficient accuracy for general use. In fact it appears that values of these constants have not as yet been determined for some of our commercial sheet metals.

The purpose of this investigation is to provide specific information concerning the effects of sizes and shapes of test specimens on the tensile properties of sheet metals of certain types and thicknesses, in order that results obtained with different test specimens may be better compared.

SCOPE OF THE INVESTIGATION

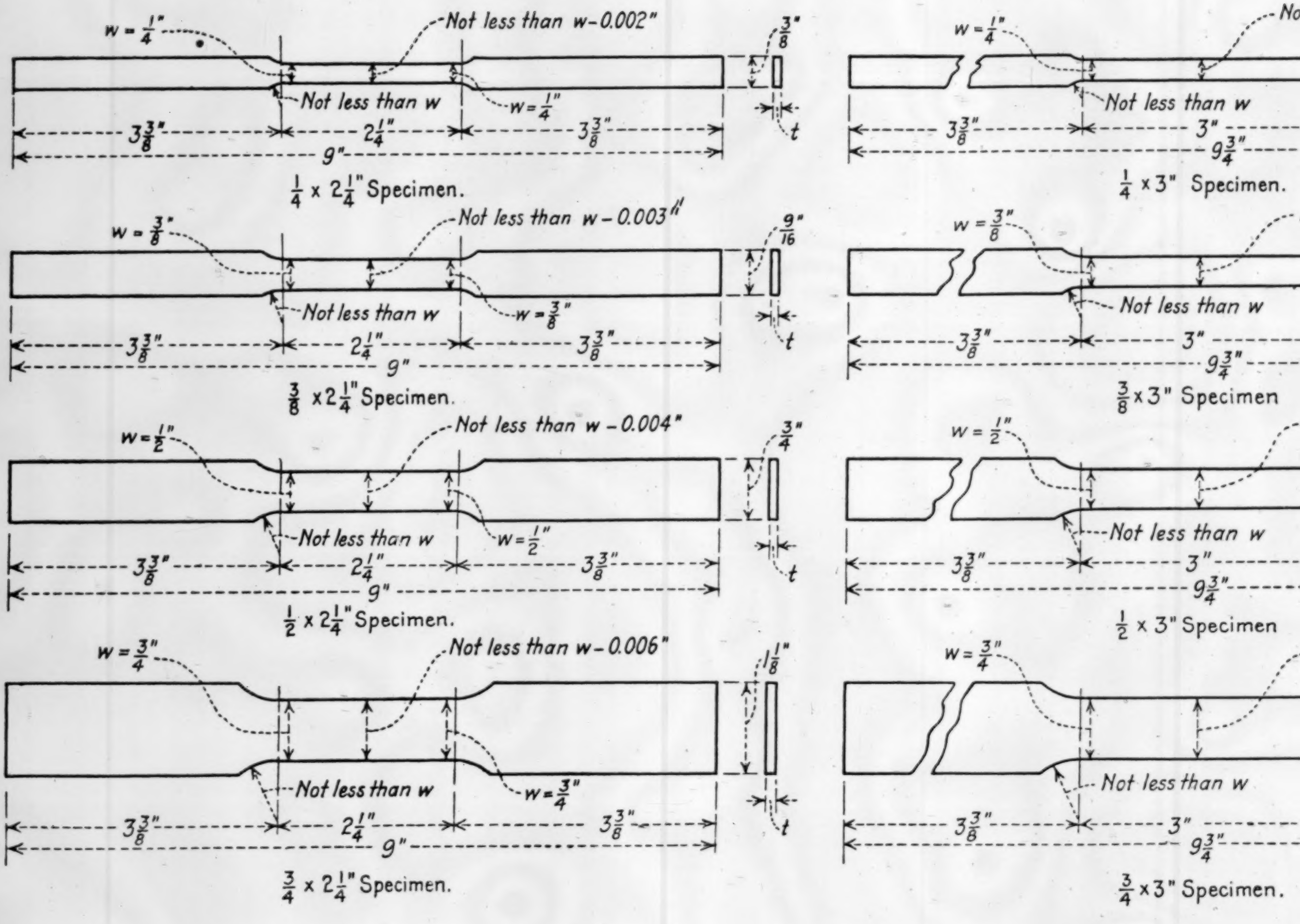
Since most of the sheet-metal tension specimens used in this country have fixed gage lengths for considerable variations in thickness of sheet, and since for the range of thickness considered, 2 in. is the gage length most used, all of the tests made in the investigation were planned on the basis of a fixed or constant gage length of 2 in. The range of specimen dimensions used was chosen so as to include many if not most of the sizes of sheet tension specimens now in use to considerable extent in this country.

The present investigation was planned to include tests on three different types of sheet metals, that is, one having a comparatively high tensile strength and low elongation (hard aluminum sheet designated as 2SH); one having a comparatively low tensile strength and high elongation (soft aluminum sheet designated as 2SO); and one having both a comparatively high tensile strength and high elongation (heat-treated duralumin designated as 17S-T). The type of material having both a comparatively low elongation and tensile strength is not usually encountered in commercial wrought sheet metals.

The first and major part of the work is concerned with the effects of variation in the thickness, width and length of reduced section of the test specimen, on the tensile properties. Throughout these tests the ends of the specimens are maintained 1.5 times the reduced section widths and the radii of fillets used are maintained equal to the reduced section widths. The details of the specimens used are shown in Fig. 2 (Plate III).

The second part of the work is concerned with the effects of variations in radius of fillets and width of ends on the tensile properties of the three types of metals indicated above. In these tests the thickness, width and length of reduced portion of the specimens are maintained constant. That is, all these specimens were cut from No. 16 B & S gage (0.051 in. thick) sheet, had a reduced section width of 0.5 in., and a reduced section length of 2.25 in. The details of these specimens are shown in Fig. 3 (Plate IV).

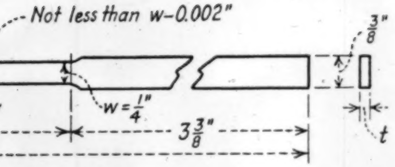
In all cases each set of conditions is represented by three test specimens, making in all a total of 774 individual tension tests in the investigation proper. Some additional tests were also made to check certain results and conclusions obtained from results of the first tests.


w = 1/4"
Not less than w - 0.002"
Not less than w
w = 1/4"
3/8"
t
3 3/8"
2 1/4"
3 3/8"
9"
1/4 x 2 1/4" Specimen.
w = 3/8"
Not less than w - 0.003"
Not less than w
w = 3/8"
9/16"
t
3 3/8"
2 1/4"
3 3/8"
9"
3/8 x 2 1/4" Specimen.
w = 1/2"
Not less than w - 0.004"
Not less than w
w = 1/2"
3/4"
t
3 3/8"
2 1/4"
3 3/8"
9"
1/2 x 2 1/4" Specimen.
w = 3/4"
Not less than w - 0.006"
Not less than w
w = 3/4"
1 1/8"
t
3 3/8"
2 1/4"
3 3/8"
9"
3/4 x 2 1/4" Specimen.
w = 1/4"
No
Not less than w
3 3/8"
3"
9 3/4"
1/4 x 3" Specimen.
w = 3/8"
Not less than w
3 3/8"
3"
9 3/4"
3/8 x 3" Specimen
w = 1/2"
Not less than w
3 3/8"
3"
9 3/4"
1/2 x 3" Specimen
w = 3/4"
Not less than w
3 3/8"
3"
9 3/4"
3/4 x 3" Specimen.

NOTE: Each of the above Specimens made in
17 S-T, 2 SO and 2SH, and in th
$\frac{1}{4}$, $\frac{1}{8}$, $\frac{1}{16}$, $\frac{1}{32}$" (No. 20 Gage) and
Specimens Prepared in Triplicate (To

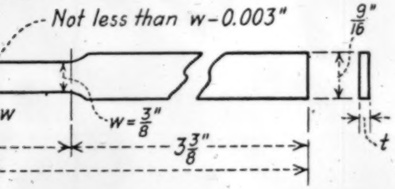
FIG. 2.—Form and Size of Tension Test

PLATE III.
AM. SOC. TESTING MATS.
VOL. 26, PART II.
TEMPLIN ON TENSION TEST SPECIMENS



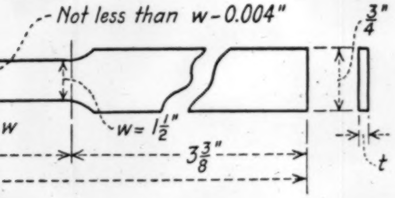
Not less than w-0.002"
3/8"
w = 1/4"
3 3/8"
t

men.



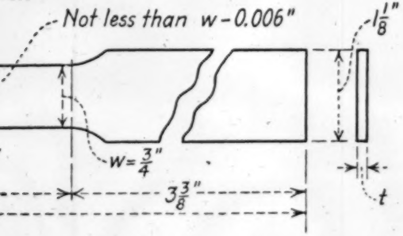
Not less than w-0.003"
9/16"
w
w = 3/8"
3 3/8"
t

nen



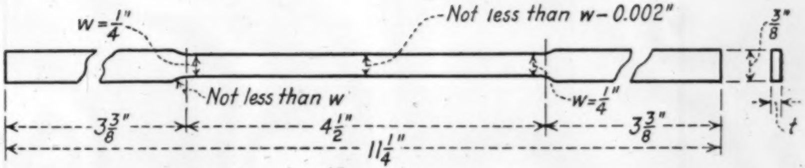
Not less than w-0.004"
3/4"
w
w = 1 1/2"
3 3/8"
t

nen



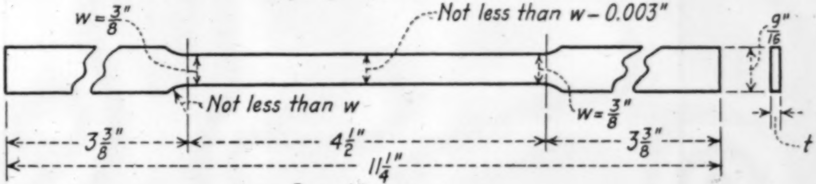
Not less than w-0.006"
1 1/8"
w = 3/4"
3 3/8"
t

nen.



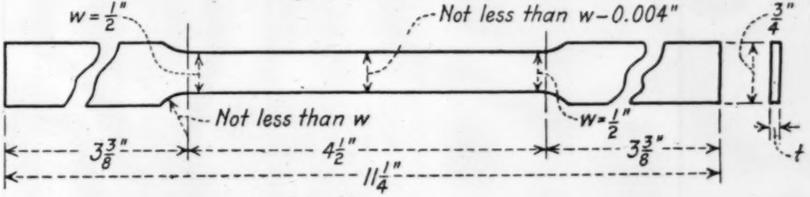
w = 1/4"
Not less than w-0.002"
3/8"
Not less than w
w = 1/4"
3 3/8"
4 1/2"
3 3/8"
11 1/4"
t

$\frac{1}{4} \times 4\frac{1}{2}$" Specimen.



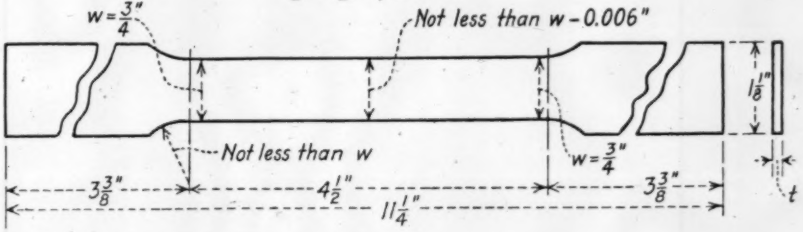
w = 3/8"
Not less than w-0.003"
9/16"
Not less than w
w = 3/8"
3 3/8"
4 1/2"
3 3/8"
11 1/4"
t

$\frac{3}{8} \times 4\frac{1}{2}$" Specimen.



w = 1/2"
Not less than w-0.004"
3/4"
Not less than w
w = 1/2"
3 3/8"
4 1/2"
3 3/8"
11 1/4"
t

$\frac{1}{2} \times 4\frac{1}{2}$" Specimen.



w = 3/4"
Not less than w-0.006"
1 1/8"
Not less than w
w = 3/4"
3 3/8"
4 1/2"
3 3/8"
11 1/4"
t

$\frac{3}{4} \times 4\frac{1}{2}$" Specimen.

ade in the Following Materials:
in the Following Thicknesses:
nd $\frac{1}{64}$" (No. 26 Gage).
: (Total, 540 Specimens).

Test Specimens for Sheet Metals.

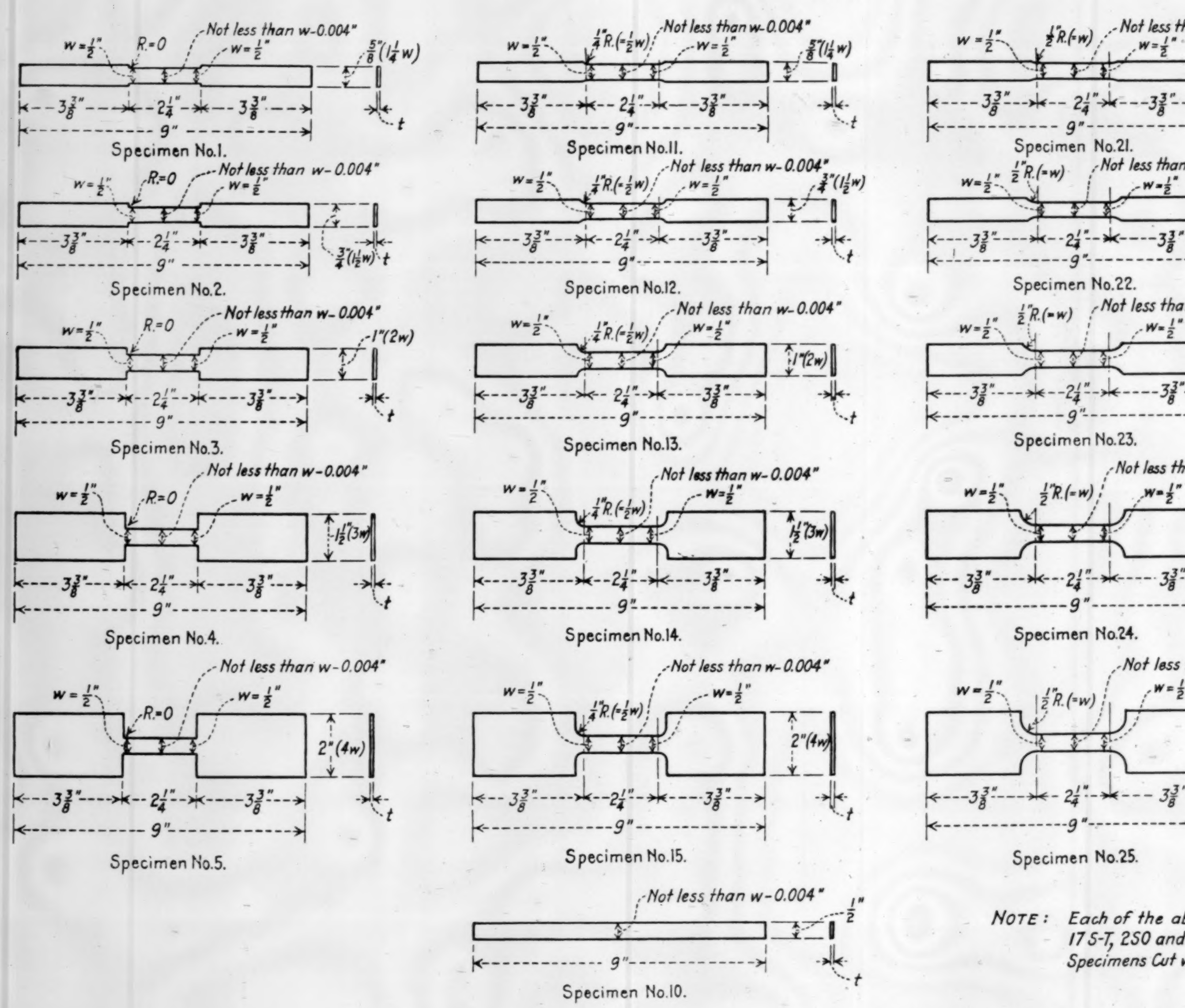
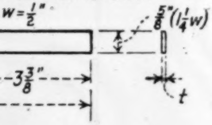
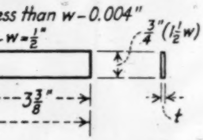
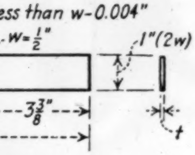

w = 1/2"
R = 0
Not less than w-0.004"
5/8"(1 1/4 w)
3 3/8"
2 1/4"
9"
t
Specimen No.1.
3/4"(1 1/2 w)
Specimen No.2.
1"(2w)
Specimen No.3.
1 1/2"(3w)
Specimen No.4.
2"(4w)
Specimen No.5.
1/4"R.(=1/2 w)
Specimen No.11.
Specimen No.12.
Specimen No.13.
Specimen No.14.
Specimen No.15.
1/2"
Specimen No.10.
1/2"R.(=w)
Specimen No.21.
Specimen No.22.
Specimen No.23.
Specimen No.24.
Specimen No.25.
NOTE: Each of the a
17S-T, 25O and
Specimens Cut

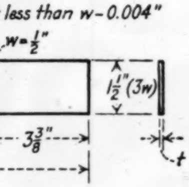
FIG. 3.—Form and Size of Tension Test Sp

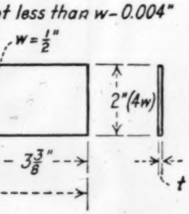
PLATE IV.
AM. SOC. TESTING MATS.
VOL. 26, PART II.
TEMPLIN ON TENSION TEST SPECIMENS

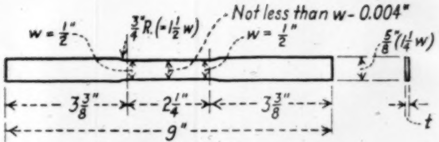

t less than w-0.004"
w = 1/2"
5/8"(1 1/4 w)
3 3/8"
t


ess than w-0.004"
w = 1/2"
3/4"(1 1/2 w)
3 3/8"
t

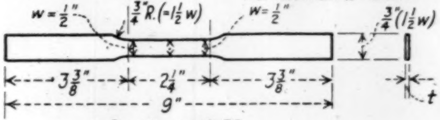

ess than w-0.004"
w = 1/2"
1"(2w)
3 3/8"
t


less than w-0.004"
w = 1/2"
1 1/2"(3w)
3 3/8"
t

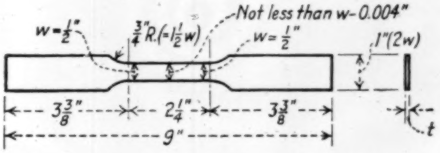

t less than w-0.004"
w = 1/2"
2"(4w)
3 3/8"
t


w = 1/2"
3/4"R.(=1 1/2 w)
Not less than w-0.004"
w = 1/2"
5/8"(1 1/4 w)
3 3/8"
2 1/4"
3 3/8"
9"
t

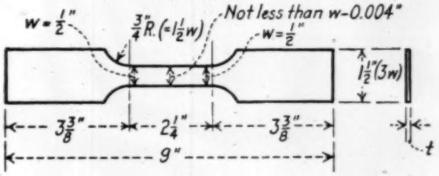
Specimen No.31.


w = 1/2"
3/4"R.(=1 1/2 w)
Not less than w-0.004"
w = 1/2"
3/4"(1 1/2 w)
3 3/8"
2 1/4"
3 3/8"
9"
t

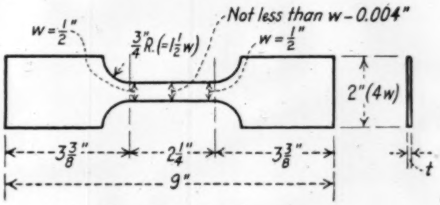
Specimen No.32.


w = 1/2"
3/4"R.(=1 1/2 w)
Not less than w-0.004"
w = 1/2"
1"(2w)
3 3/8"
2 1/4"
3 3/8"
9"
t

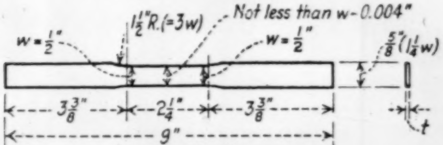
Specimen No.33.


w = 1/2"
3/4"R.(=1 1/2 w)
Not less than w-0.004"
w = 1/2"
1 1/2"(3w)
3 3/8"
2 1/4"
3 3/8"
9"
t

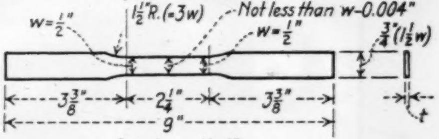
Specimen No.34.


w = 1/2"
3/4"R.(=1 1/2 w)
Not less than w-0.004"
w = 1/2"
2"(4w)
3 3/8"
2 1/4"
3 3/8"
9"
t

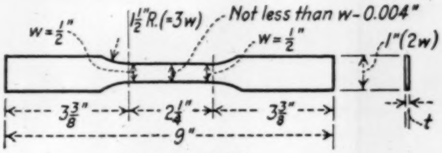
Specimen No.35.


w = 1/2"
1 1/2"R.(=3w)
Not less than w-0.004"
w = 1/2"
5/8"(1 1/4 w)
3 3/8"
2 1/4"
3 3/8"
9"
t

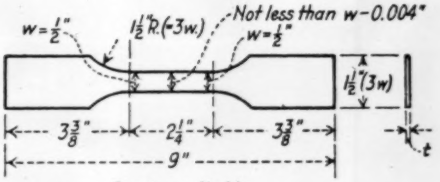
Specimen No.41.


w = 1/2"
1 1/2"R.(=3w)
Not less than w-0.004"
w = 1/2"
3/4"(1 1/2 w)
3 3/8"
2 1/4"
3 3/8"
9"
t

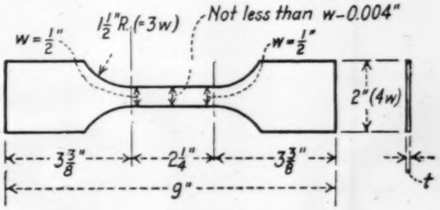
Specimen No.42.


w = 1/2"
1 1/2"R.(=3w)
Not less than w-0.004"
w = 1/2"
1"(2w)
3 3/8"
2 1/4"
3 3/8"
9"
t

Specimen No.43.


w = 1/2"
1 1/2"R.(=3w.)
Not less than w-0.004"
w = 1/2"
1 1/2"(3w)
3 3/8"
2 1/4"
3 3/8"
9"
t

Specimen No.44.


w = 1/2"
1 1/2"R.(=3w)
Not less than w-0.004"
w = 1/2"
2"(4w)
3 3/8"
2 1/4"
3 3/8"
9"
t

Specimen No.45.

the above Specimens made in the Following Materials:
0 and 2 SH, and in Thickness (t) B & S Gage No. 16 (0.051").
s Cut with Grain and in Triplicate (Total 234 Specimens).

est Specimens for Sheet Metals.

PREPARATION AND TESTING OF SPECIMENS

With the exception of specimens Nos. 1 to 5, inclusive, shown in Fig. 3, all of the specimens were prepared by sawing or shearing oversize blanks from the sheet metal used, then milling the specimens in a direction parallel to their longitudinal axes, to the dimensions indicated. Specimens Nos. 1 to 5 were milled normal to their longitudinal axes, then scraped to the dimensions indicated to remove any transverse cutter marks or scratches. In all cases where the specimen blanks were sheared, care was taken to see that an amount of metal equal to or greater than the thickness of the sheet from which the specimens were cut was removed by milling from the edges of the specimens throughout their lengths.

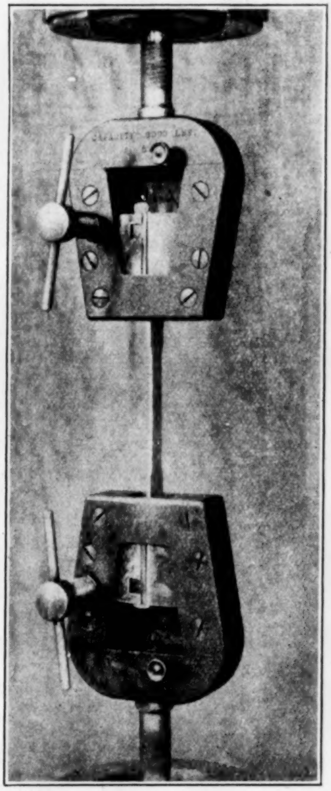


FIG. 4.—Showing Test Specimen in Position in the Holders of the Testing Machine.

Two or more gage lengths were put on each specimen by means of prick punch marks placed on the center line of the specimen and staggered distances varying from $\frac{1}{4}$ to $\frac{1}{2}$ in. depending on the length of reduced section.

TABLE I.—EFFECT OF SIZE AND SHAPE OF REDUCED SECTION ON THE TENSILE PROPERTIES OF ALUMINUM SHEET.

All results are average of 3 tests.

SOFT ALUMINUM SHEET—2SO

Length Exclusive of Fillets, in.	Nominal Width, in.	Actual Thickness, in.	Ratio of Width to Thickness	Cross-Sectional Area, sq. in.	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent
2¼	0.250	0.2480	1.01	0.0615	12 940	4 900	37.2
	0.250	0.1255	1.99	0.0307	12 980	4 600	34.3
	0.250	0.0635	3.94	0.0158	12 980	5 100	31.8
	0.250	0.0305	8.20	0.00747	12 940	5 100	31.0
	0.250	0.0160	15.63	0.00394	12 360	4 900	26.8
2¼	0.375	0.2430	1.54	0.0909	13 370	4 100	39.7
	0.375	0.1260	2.98	0.0472	12 870	4 000	35.0
	0.375	0.0630	5.95	0.0236	12 960	4 800	33.0
	0.375	0.0305	12.30	0.0114	12 870	4 900	33.3
	0.375	0.0155	24.19	0.00581	12 460	4 600	30.5
2¼	0.500	0.2470	2.02	0.1227	13 120	3 900	40.7
	0.500	0.1255	3.98	0.0622	13 030	4 600	36.8
	0.500	0.0630	7.94	0.0313	13 110	5 000	34.8
	0.500	0.0305	16.40	0.0151	12 860	4 500	33.7
	0.500	0.0155	32.26	0.00770	12 900	4 700	31.2
2¼	0.750	0.2470	3.04	0.1853	13 050	4 300	43.5
	0.750	0.1255	5.98	0.0942	13 020	4 800	40.0
	0.750	0.0630	11.91	0.0473	12 910	4 900	37.8
	0.750	0.0300	25.00	0.0225	12 980	4 600	36.5
	0.750	0.0150	50.00	0.0113	12 870	5 200	33.2
3	0.250	0.2480	1.01	0.0615	13 150	5 000	37.5
	0.250	0.1252	2.00	0.0312	12 900	4 500	33.2
	0.250	0.0638	3.92	0.0158	13 000	4 900	33.3
	0.250	0.0305	8.20	0.00756	12 860	5 200	31.5
	0.250	0.0157	15.92	0.00388	12 630	5 100	29.2
3	0.375	0.2480	1.51	0.0928	13 080	3 900	40.8
	0.375	0.1250	3.00	0.0468	12 950	4 400	35.0
	0.375	0.0638	5.88	0.0237	12 850	5 000	34.5
	0.375	0.0305	12.30	0.0114	12 880	5 000	34.0
	0.375	0.0155	24.19	0.00577	12 780	4 700	30.5
3	0.500	0.2480	2.02	0.1235	13 090	3 900	43.0
	0.500	0.1250	4.00	0.0622	13 050	4 700	38.3
	0.500	0.0638	7.84	0.0316	13 080	4 900	37.0
	0.500	0.0300	16.67	0.0149	13 010	4 700	36.0
	0.500	0.0153	32.68	0.00764	12 920	4 800	32.2
3	0.750	0.2480	3.02	0.1857	12 970	4 200	43.7
	0.750	0.1250	6.00	0.0938	12 950	4 500	39.7
	0.750	0.0640	11.72	0.0478	12 820	4 900	39.3
	0.750	0.0302	24.84	0.0226	12 900	5 000	37.7
	0.750	0.0150	50.00	0.0112	12 950	5 200	34.3
4½	0.250	0.2475	1.01	0.0611	13 030	5 000	39.0
	0.250	0.1245	2.01	0.0304	12 990	4 600	36.0
	0.250	0.0635	3.94	0.0156	13 030	5 100	35.0
	0.250	0.0300	8.33	0.00733	13 010	4 900	34.3
	0.250	0.0155	16.13	0.00381	12 680	5 100	30.3
4½	0.375	0.2480	1.51	0.0922	13 080	3 700	41.0
	0.375	0.1250	3.00	0.0465	12 880	4 600	37.0
	0.375	0.0640	5.86	0.0237	12 820	4 800	36.3
	0.375	0.0303	12.38	0.0113	12 770	4 500	34.5
	0.375	0.0160	23.44	0.00595	12 430	4 300	31.7
4½	0.500	0.2480	2.02	0.1239	13 100	4 200	42.7
	0.500	0.1250	4.00	0.0622	12 970	4 200	37.7
	0.500	0.0640	7.81	0.0319	13 020	4 600	38.0
	0.500	0.0300	16.67	0.0149	13 010	4 600	36.7
	0.500	0.0155	32.26	0.00774	12 960	4 600	33.5
4½	0.750	0.2473	3.03	0.1852	12 950	4 400	44.3
	0.750	0.1248	6.01	0.0937	12 970	4 000	41.5
	0.750	0.0640	11.72	0.0481	12 770	4 600	40.5
	0.750	0.0300	25.00	0.0225	12 850	4 500	39.7
	0.750	0.0160	46.88	0.0120	12 450	4 500	35.2

TABLE I.—EFFECT OF SIZE AND SHAPE OF REDUCED SECTION ON THE TENSILE PROPERTIES OF ALUMINUM SHEET (*Continued*).

HARD ALUMINUM SHEET—2SH

Length Exclusive of Fillets, in.	Nominal Width, in.	Actual Thickness, in.	Ratio of Width to Thickness	Cross-Sectional Area, sq. in.	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent
2¼	0.250	0.2483	1.01	0.0608	21 920	19 900	11.2
	0.250	0.1253	2.00	0.0305	22 950	21 300	7.33
	0.250	0.0645	3.88	0.0156	21 830	19 200	6.33
	0.250	0.0308	8.12	0.00750	21 370	19 600	3.83
	0.250	0.0155	16.13	0.00381	22 470	20 400	2.83
2¼	0.375	0.2480	1.51	0.0932	21 890	19 700	11.2
	0.375	0.1255	2.99	0.0472	23 160	20 800	7.67
	0.375	0.0645	5.81	0.0242	21 890	19 500	7.33
	0.375	0.0307	12.22	0.0115	21 250	19 500	5.00
	0.375	0.0155	24.19	0.00580	22 530	20 900	3.00
2¼	0.500	0.2480	2.02	0.1234	21 940	20 100	10.8
	0.500	0.1255	3.98	0.0623	22 960	21 100	8.50
	0.500	0.0645	7.75	0.0320	21 790	19 200	7.50
	0.500	0.0300	16.67	0.0149	21 860	19 900	4.67
	0.500	0.0150	33.33	0.00746	22 750	20 500	3.50
2¼	0.750	0.2480	3.02	0.1858	22 470	20 100	11.0
	0.750	0.1255	5.98	0.0941	23 030	20 200	8.83
	0.750	0.0640	11.72	0.0479	22 260	19 800	7.83
	0.750	0.0300	25.00	0.0225	21 840	19 400	5.50
	0.750	0.0150	50.00	0.0113	22 690	20 000	4.50
3	0.250	0.2478	1.01	0.0626	21 810	19 600	11.7
	0.250	0.1255	1.99	0.0314	23 100	21 200	7.67
	0.250	0.0647	3.86	0.0161	21 850	18 700	6.83
	0.250	0.0300	8.33	0.00746	21 900	19 700	4.17
	0.250	0.0155	16.13	0.00386	22 480	20 000	3.17
3	0.375	0.2475	1.52	0.0930	21 900	19 400	11.8
	0.375	0.1250	3.00	0.0468	23 400	21 000	7.83
	0.375	0.0650	5.77	0.0243	21 800	19 100	7.50
	0.375	0.0300	12.50	0.0112	21 660	19 700	5.17
	0.375	0.0152	24.67	0.00561	23 060	20 800	3.33
3	0.500	0.2475	2.02	0.1234	21 990	20 100	11.2
	0.500	0.1250	4.00	0.0623	23 130	21 300	8.50
	0.500	0.0650	7.69	0.0324	21 700	19 600	8.00
	0.500	0.0300	16.67	0.0149	21 900	19 800	5.33
	0.500	0.0150	33.33	0.00748	22 960	20 500	3.67
3	0.750	0.2475	3.03	0.1852	22 220	20 000	11.5
	0.750	0.1245	6.02	0.0932	23 230	20 400	8.50
	0.750	0.0647	11.60	0.0484	22 060	19 400	8.33
	0.750	0.0300	25.00	0.0224	21 890	18 900	6.00
	0.750	0.0150	50.00	0.0112	22 740	20 400	4.25
4½	0.250	0.2480	1.01	0.0606	21 840	19 900	11.5
	0.250	0.1250	2.00	0.0305	22 980	21 200	7.50
	0.250	0.0655	3.82	0.0160	21 560	18 900	6.50
	0.250	0.0303	8.25	0.00741	21 550	19 300	4.17
	0.250	0.0160	15.63	0.00392	21 840	19 100	3.50
4½	0.375	0.2480	1.51	0.0927	21 820	19 000	11.8
	0.375	0.1250	3.00	0.0465	23 220	20 400	8.50
	0.365	0.0655	5.73	0.0243	21 660	19 000	7.50
	0.375	0.0300	12.50	0.0112	21 640	19 600	4.67
	0.375	0.0157	23.89	0.00582	22 290	19 700	3.83
4½	0.500	0.2480	2.02	0.1234	21 970	20 000	11.3
	0.500	0.1250	4.00	0.0623	22 900	20 900	9.33
	0.500	0.0655	7.63	0.0327	21 530	19 100	8.17
	0.500	0.0300	16.67	0.0150	21 670	19 500	4.83
	0.500	0.0158	31.64	0.00782	21 800	19 400	3.83
4½	0.750	0.2475	3.03	0.1859	22 410	19 900	12.0
	0.750	0.1250	6.00	0.0938	23 000	20 100	10.3
	0.750	0.0655	11.45	0.0491	21 710	19 100	8.50
	0.750	0.0300	25.00	0.0225	21 700	19 000	5.33
	0.750	0.0155	48.39	0.0116	22 150	19 800	4.17

TABLE I.—EFFECT OF SIZE AND SHAPE OF REDUCED SECTION ON THE TENSILE PROPERTIES OF ALUMINUM SHEET (*Continued*).

HEAT-TREATED DURALUMIN SHEET—17S - T

Length Exclusive of Fillets, in.	Nominal Width, in.	Actual Thickness, in.	Ratio of Width to Thickness	Cross-Sectional Area, sq. in.	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent
2¼	0.250	0.2500	1.00	0.0622	60 640	38 700	20.2
	0.250	0.1245	2.01	0.0307	62 560	43 200	18.3
	0.250	0.0635	3.94	0.0157	60 640	42 200	17.8
	0.250	0.0310	8.06	0.00767	59 420	41 700	17.7
	0.250	0.0165	15.15	0.00407	59 680	40 500	16.7
2¼	0.375	0.2505	1.50	0.0938	60 650	39 200	21.5
	0.375	0.1245	3.01	0.0467	62 560	42 800	20.7
	0.375	0.0640	5.86	0.0240	61 400	41 800	19.0
	0.375	0.0315	11.91	0.0118	58 680	41 500	18.5
	0.375	0.0170	22.06	0.00637	57 150	39 000	17.5
2¼	0.500	0.2505	2.00	0.1252	60 070	39 300	22.2
	0.500	0.1240	4.03	0.0619	62 290	42 800	21.0
	0.500	0.0643	7.78	0.0320	60 530	41 600	18.9
	0.500	0.0315	15.87	0.0157	58 710	42 000	18.0
	0.500	0.0170	29.41	0.00846	57 500	38 200	18.7
2¼	0.750	0.2510	2.99	0.1886	59 930	38 300	22.3
	0.750	0.1240	6.05	0.0932	62 100	41 500	22.8
	0.750	0.0640	11.72	0.0480	61 350	42 700	21.3
	0.750	0.0315	23.82	0.0237	58 560	42 000	16.7
	0.750	0.0165	45.46	0.0124	59 170	40 500	19.2
3	0.250	0.2515	0.99	0.0623	60 140	38 000	21.2
	0.250	0.1240	2.02	0.0307	61 380	42 800	19.8
	0.250	0.0630	3.97	0.0156	60 820	42 000	18.8
	0.250	0.0315	7.94	0.00778	59 080	41 200	18.2
	0.250	0.0170	14.71	0.00420	58 290	39 300	17.7
3	0.375	0.2510	1.49	0.0941	59 800	38 200	21.0
	0.375	0.1245	3.01	0.0464	61 670	42 300	21.0
	0.375	0.0638	5.88	0.0238	60 780	41 700	19.5
	0.375	0.0315	11.90	0.0117	59 460	41 800	19.0
	0.375	0.0170	22.06	0.00635	56 930	38 800	17.2
3	0.500	0.2510	1.99	0.1252	59 150	37 700	22.3
	0.500	0.1240	4.03	0.0620	61 650	42 500	22.5
	0.500	0.0633	7.90	0.0316	60 240	41 800	19.3
	0.500	0.0310	16.13	0.0155	59 560	42 200	19.5
	0.500	0.0168	29.76	0.00839	59 140	40 200	19.3
3	0.750	0.2498	3.00	0.1872	59 800	37 200	23.5
	0.750	0.1238	6.06	0.0928	61 650	41 500	23.7
	0.750	0.0635	11.81	0.0476	61 080	42 700	21.0
	0.750	0.0315	23.81	0.0236	58 710	42 300	18.8
	0.750	0.0165	45.46	0.0123	60 060	40 500	20.2
4½	0.250	0.2510	1.00	0.0624	59 160	36 500	20.2
	0.250	0.1245	2.01	0.0309	60 750	41 500	20.0
	0.250	0.0638	3.92	0.0157	60 050	41 300	17.8
	0.250	0.0310	8.06	0.00764	59 590	41 500	17.8
	0.250	0.0170	14.70	0.00418	58 980	40 000	17.5
4½	0.375	0.2512	1.49	0.0943	58 950	36 000	21.5
	0.375	0.1245	3.01	0.0464	60 930	41 700	21.3
	0.375	0.0625	5.86	0.0238	60 890	41 800	19.2
	0.375	0.0310	12.10	0.0115	59 310	43 000	19.3
	0.375	0.0170	22.06	0.00632	58 420	39 500	19.3
4½	0.500	0.2513	1.99	0.1254	59 310	36 000	20.8
	0.500	0.1245	4.02	0.0623	60 730	42 000	21.7
	0.500	0.0638	7.84	0.0319	60 090	41 700	19.5
	0.500	0.0315	15.87	0.0157	58 590	42 800	19.8
	0.500	0.0170	29.41	0.00851	58 530	39 200	19.8
4½	0.750	0.2510	2.99	0.1882	59 770	36 300	22.3
	0.750	0.1238	6.06	0.0929	61 690	41 200	23.8
	0.750	0.0638	11.76	0.0480	60 990	42 000	22.2
	0.750	0.0310	24.20	0.0233	59 540	44 000	19.2
	0.750	0.0170	44.12	0.0127	59 550	40 000	20.0

The specimens were all tested using an Amsler testing machine having seven different sensitivities and capacities ranging from 200 to 20,000 lb. For the specimens whose ends were 0.75 in. or less in width special grips were used. These are shown in Fig. 4. For the larger specimens, wedge grips similar to those usually furnished with the testing machine but having finer double-cut teeth were used. The use of special grips usually will be found necessary in the successful tension testing of thin sheet metals.

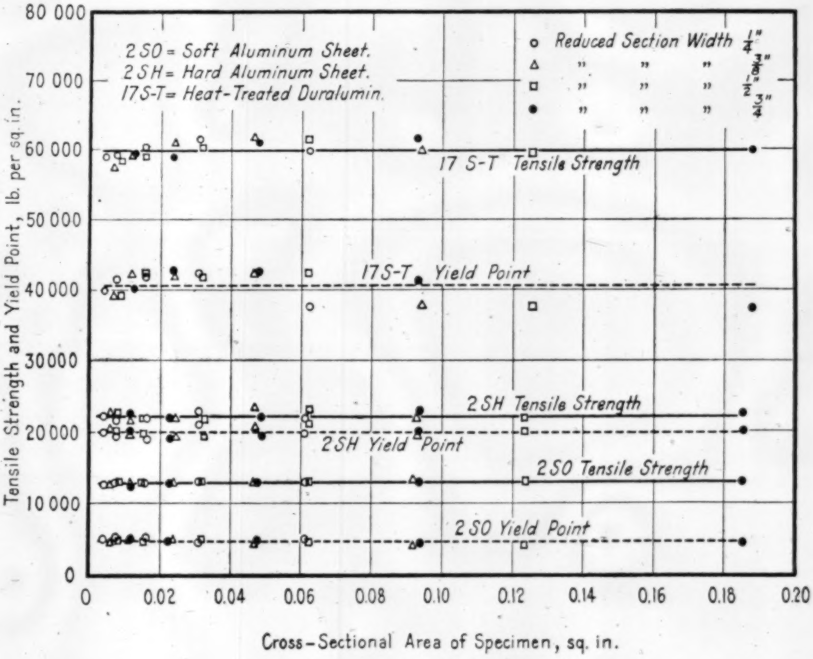


FIG. 5.—Showing Relation of Tensile Strength and Yield Point to the Cross-Sectional Area of Specimen.

The yield point values indicated in the accompanying figures and tables correspond closely to the unit stress at which the unit deformation under load is equal to 0.5 per cent.

EFFECTS OF SIZE AND SHAPE OF REDUCED SECTION ON THE TENSILE PROPERTIES

The results of tests made for determining the effects of size and shape of reduced section on the tensile properties of the three types of sheet metal are given in Table I.

Effect on Tensile Strength and Yield Point:

In Fig. 5, the tensile strength and yield point values have been plotted against the cross-sectional areas of the specimens, using different symbols for different widths of reduced section, in order to show first, the absence of any effects on these properties of variations in the reduced section dimensions of test specimens; and secondly, the uniformity of material used for this part of the investigation. From these data we are led to conclude that for variations in the width to thickness ratio w/t, of from 1 to 45; for cross-sectional areas from 0.004 sq. in. to 0.188 sq. in.; and for length of reduced sections from 2.25 to 4.50 in., no effects on the tensile strength and yield point values are observed, as might be expected.

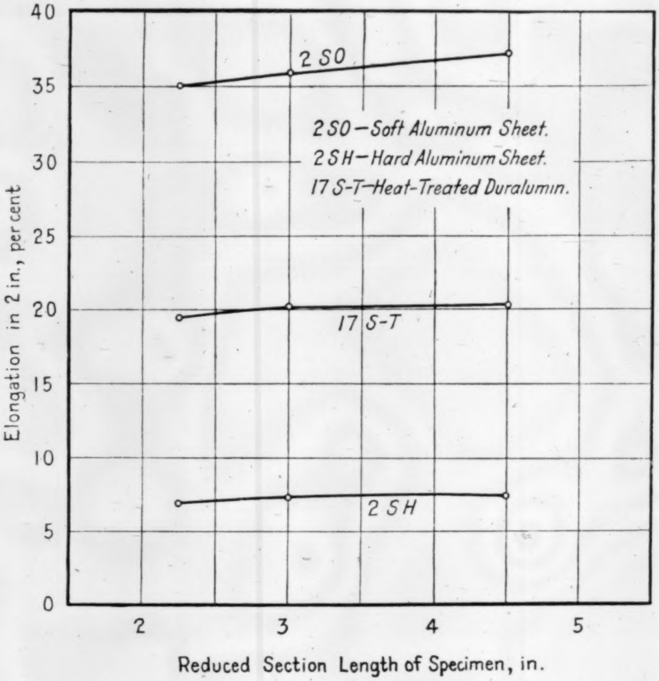


FIG. 6.—Showing Relation Between Elongation and the Length of Reduced Section.

Effect on Elongation:

The effects of variations in the reduced section on the elongation using a fixed gage length of 2 in., however, are quite noticeable. The elongation values from all the specimens are first plotted with respect to type of metal against the length of reduced section as shown in

Fig. 6. The curves obtained indicate that there is a slight increase in elongation in each case with increase in length of reduced section. This is in agreement with similar results obtained by other investigators. It may be further noted that the percentage increase in the elongation values is about the same for each type of material tested.

When the elongation values are plotted against the width of reduced section, again with respect to type of material, we obtain the curves shown in Fig. 7, and when plotted against thickness of material we obtain the curves shown in Fig. 8. Both Figs. 7 and 8 indicate that the cross-sectional dimensions have an appreciable effect on the elongation as measured over a fixed gage length. Heretofore many investigators have plotted the elongation values against the ratio of w/t (width to thickness of specimen). This procedure, however, often gives graphs whose interpretation leads to erroneous conclusions such as the value of the ratio affecting elongation differently for different materials or certain critical values of the ratio giving maximum elongation values.

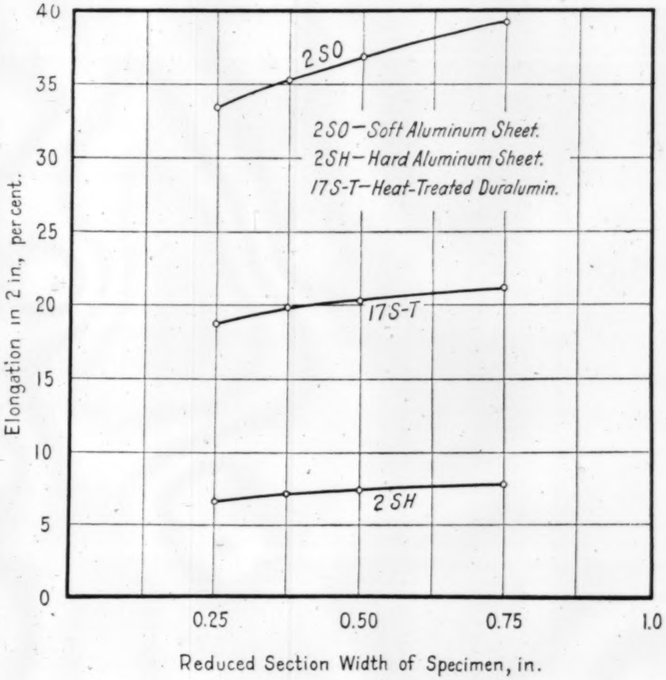


FIG. 7.—Showing Relation Between Elongation and the Width of Reduced Section.

Plotting the elongation values obtained in this investigation on a fixed gage length of 2 in., against the total cross-sectional areas of the reduced sections of the specimens gives curves as shown in Fig. 9. The ratio values of w/t for the 17S-T curve points have been indicated on the graph and opposite points with corresponding symbols on the other two curves have the same w/t values. A study of the w/t values as shown in Fig. 9 indicates that this ratio between values of 1 and 45 has little if any effect on the elongation values shown. This has been found true also for values of w/t up to 60 from results obtained by other investigators, although not emphasized or in some cases not recognized by them.

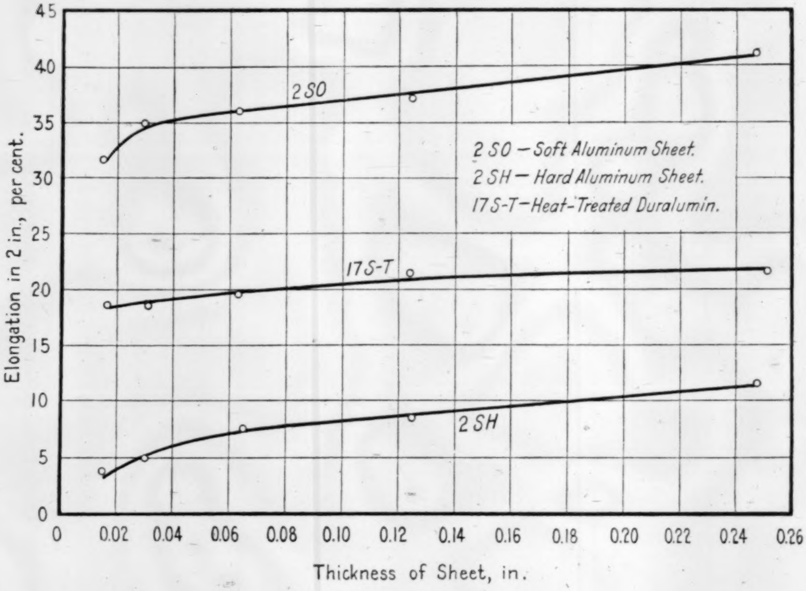


FIG. 8.—Showing Relation Between Elongation and the Thickness of Sheet.

Replotting the data shown in Fig. 9 on logarithmic coordinates, we obtain the curves shown in Fig. 10, indicating that the elongation - cross-sectional area relationship may be expressed by the general equation:

$$e_2 = KA^n \qquad (1)$$

where, e_2 = per cent of elongation in 2 in., A = the cross-sectional area of the reduced portions of the specimens, and K and n are constants.

For the three curves shown, the constants have approximately the following values:

For the 2SO.................................. $e_2 = 52A^{0.1}$
For the 17S-T................................ $e_2 = 26A^{0.07}$.
For the 2SH.................................. $e_2 = 25A^{0.37}$

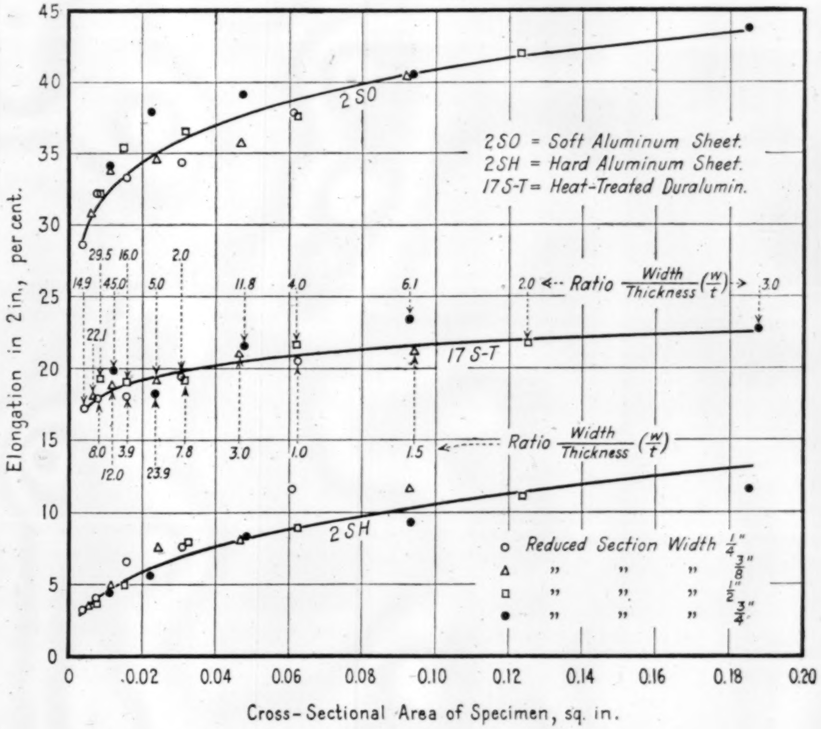


FIG. 9.—Showing Relation Between Elongation and Cross-Sectional Area of Specimen.

Similar data obtained from Gordon's (**16**)[1] results and Beare and Gordon's (**18**) results give for mild steel (0.138 in. and 0.125 in. thick using specimens 0.250 in. to 4 in. wide):

$$e_2 = 46A^{0.1},$$

and for annealed copper (0.125 in. thick using specimens 0.250 in. to 4 in. wide):

$$e_2 = 68A^{0.1},$$

the symbols having the same meaning as above. Again we obtain the equation for soft annealed brass (66 per cent copper, 33 per cent zinc) using thicknesses from 0.010 in. to 0.066 in., from Davis' (19) results,

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[1] The boldface numbers in parentheses refer to the papers given in the list of references appended hereto.

$$e_2 = 80A^{0.1}.$$

These three metals having comparatively low tensile strengths and high elongations are comparable to the 2SO metal herein discussed. The value of 0.1 for n observed in each case would appear significant in that it suggests that metals of a similar type will show elongation values, when using a fixed gage length, which are affected in the same manner by variations in cross-sectional area of the specimen. Further data concerning the value of this constant, however, would seem to be necessary before accepting the value indicated.

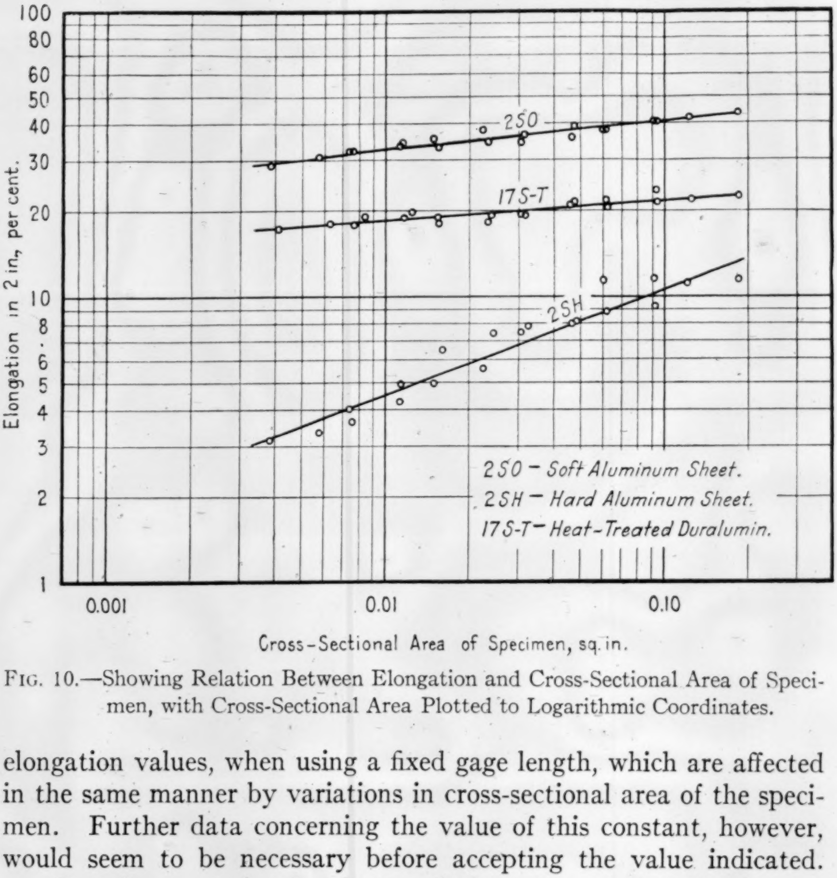


FIG. 10.—Showing Relation Between Elongation and Cross-Sectional Area of Specimen, with Cross-Sectional Area Plotted to Logarithmic Coordinates.

Bertella (17), using specimens of the same cross-sectional area, has shown the effects of variations in the ratio of gage length to square root of cross-sectional area on the elongation of various metals, and in so doing develops the general equation for elongation as follows:

$$e = K' \left(\frac{L}{\sqrt{A}}\right)^n \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

TABLE II.—EFFECT OF SHAPE OF CROSS-SECTION ON THE TENSILE PROPERTIES OF ALUMINUM SHEET (2SO) WITH CROSS-SECTIONAL AREA KEPT CONSTANT.

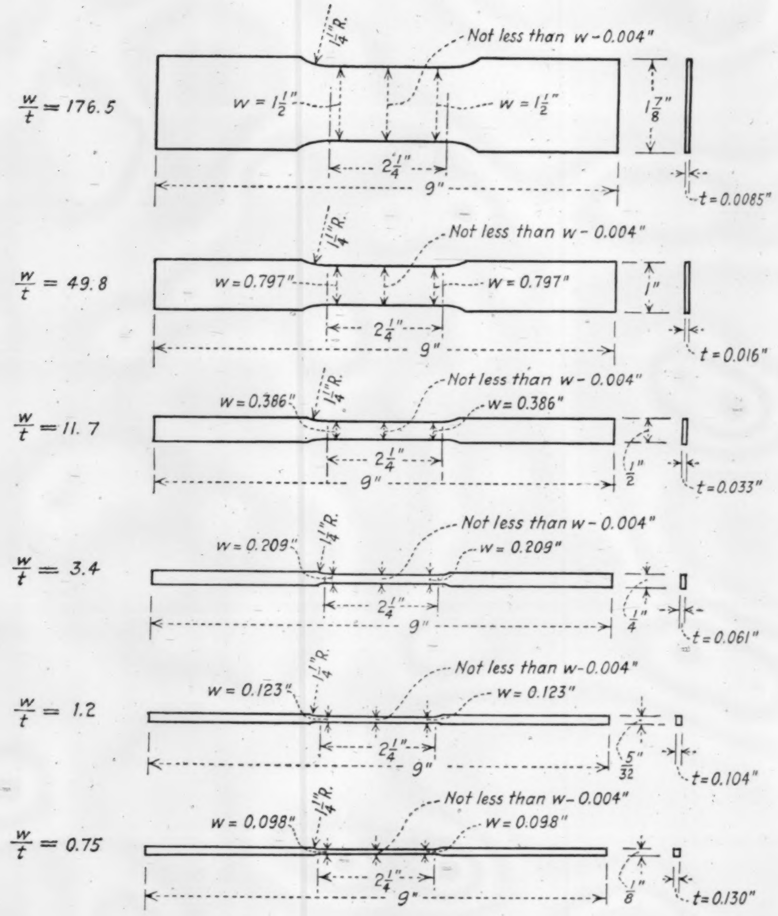
All results are average of 3 tests.

Thickness, in.	Width, in.	Cross-Sectional Area, sq. in.	Ratio of Width to Thickness	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent
0.0085	1.500	0.01275	176.5	13 650	5 500	27.3
0.0160	0.794	0.01270	49.6	13 660	6 750	29.0
0.0330	0.382	0.01261	11.6	13 565	5 750	30.0
0.0610	0.203	0.01238	3.3	13 685	6 750	33.5
0.1043	0.123	0.01283	1.2	14 675	6 750	25.7
0.1300	0.097	0.01261	0.75	13 595	7 000	29.3

TABLE III.—EFFECT OF SIZE AND SHAPE OF SPECIMEN ENDS ON THE TENSILE PROPERTIES OF ALUMINUM SHEET.

All results are average of 3 tests.

Specimen Number	Width of Ends, in.	Radius of Fillets, in.	Soft Aluminum Sheet—2SO			Hard Aluminum Sheet—2SH			Heat-Treated Duralumin Sheet—17S - T		
			Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent
1	$\frac{5}{8}$	0	12 315	5 700	27.8	30 650	24 800	4.00	55 630	41 750	7.17
2	$\frac{3}{4}$	0	12 365	5 300	25.2	28 600	23 700	4.00	56 575	42 500	7.33
3	1	0	12 355	6 450	23.0	29 175	24 800	4.00	55 890	42 750	6.50
4	$1\frac{1}{2}$	0	12 455	6 450	20.5	29 210	25 000	4.00	54 630	42 500	6.00
5	2	0	12 105	6 550	19.3	29 655	24 900	4.00	55 870	43 250	6.83
11	$\frac{5}{8}$	$\frac{1}{4}$	11 965	6 100	34.2	29 255	25 800	4.33	59 745	43 200	17.8
12	$\frac{3}{4}$	$\frac{1}{4}$	12 105	5 300	35.8	29 250	24 700	4.00	59 430	42 300	17.8
13	1	$\frac{1}{4}$	12 230	5 500	34.8	29 285	24 900	3.83	59 865	40 800	18.0
14	$1\frac{1}{2}$	$\frac{1}{4}$	12 180	5 100	37.0	29 075	26 900	3.67	59 745	43 000	18.2
15	2	$\frac{1}{4}$	12 505	5 400	34.5	28 645	25 900	3.83	59 805	43 000	18.3
21	$\frac{5}{8}$	$\frac{1}{2}$	12 035	5 700	33.7	30 100	27 500	4.00	59 645	43 000	18.8
22	$\frac{3}{4}$	$\frac{1}{2}$	12 255	5 200	35.2	28 980	24 700	4.00	59 655	42 800	17.2
23	1	$\frac{1}{2}$	12 285	5 900	33.8	29 435	24 900	4.00	59 685	41 500	17.7
24	$1\frac{1}{2}$	$\frac{1}{2}$	12 220	5 100	35.5	29 160	26 000	3.67	60 205	43 500	18.0
25	2	$\frac{1}{2}$	12 410	5 200	35.0	29 165	26 300	4.00	60 270	42 000	19.2
31	$\frac{5}{8}$	$\frac{3}{4}$	12 195	5 600	34.7	29 195	26 300	4.33	59 895	42 700	17.7
32	$\frac{3}{4}$	$\frac{3}{4}$	12 115	4 900	35.7	29 355	25 100	4.00	59 495	42.500	17.2
33	1	$\frac{3}{4}$	12 155	5 100	34.2	29 370	25 500	4.00	59 610	42 000	17.2
34	$1\frac{1}{2}$	$\frac{3}{4}$	12 330	4 900	35.9	30 020	26 800	4.00	60 230	41 700	18.5
35	2	$\frac{3}{4}$	12 225	4 800	35.2	29 300	26 400	4.00	60 380	43 000	18.3
41	$\frac{5}{8}$	$1\frac{1}{2}$	12 090	5 300	34.5	28 910	25 400	4.00	60 135	42 500	17.7
42	$\frac{3}{4}$	$1\frac{1}{2}$	12 145	5 000	31.7	28 665	25 800	4.00	60 070	43 500	17.7
43	1	$1\frac{1}{2}$	12 440	5 500	34.7	28 925	26 200	3.83	59 590	41 500	18.5
44	$1\frac{1}{2}$	$1\frac{1}{2}$	12 265	5 000	33.0	29 585	25 800	4.00	60 305	41 800	15.5
45	2	$1\frac{1}{2}$	12 270	4 800	31.5	29 825	25 300	4.17	59 735	42 500	16.2
10	$\frac{1}{2}$	∝	12 275	5 600	34.3	30 020	23 800	4.00	59 765	42 500	20.5


$\frac{w}{t} = 176.5$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = $1\frac{1}{2}$"
w = $1\frac{1}{2}$"
$1\frac{7}{8}$"
$2\frac{1}{4}$"
9"
t = 0.0085"
$\frac{w}{t} = 49.8$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = 0.797"
w = 0.797"
1"
$2\frac{1}{4}$"
9"
t = 0.016"
$\frac{w}{t} = 11.7$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = 0.386"
w = 0.386"
$\frac{1}{2}$"
$2\frac{1}{4}$"
9"
t = 0.033"
$\frac{w}{t} = 3.4$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = 0.209"
w = 0.209"
$\frac{1}{4}$"
$2\frac{1}{4}$"
9"
t = 0.061"
$\frac{w}{t} = 1.2$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = 0.123"
w = 0.123"
$\frac{5}{32}$"
$2\frac{1}{4}$"
9"
t = 0.104"
$\frac{w}{t} = 0.75$
$1\frac{1}{4}$" R.
Not less than w - 0.004"
w = 0.098"
w = 0.098"
$\frac{1}{8}$"
$2\frac{1}{4}$"
9"
t = 0.130"

Note:
Each of the above Specimens are to be Cut from the Indicated Thicknesses of 2SO Sheet. The Specimens are to be cut with Grain and in Triplicate. (Total 18 Specimens.)
The Cross-Sectional Area of each Specimen is approximately 0.01275 sq. in. Elongation to be Measured over a 2-in. Gage Length.

FIG. 11.—Tension Test Specimens.

where e = per cent of elongation in any gage length, K' = a coefficient varying with the elongation, L = the gage length, A = cross-sectional area of the reduced portion of the specimen, and n = a constant.

Bertella's equation is based upon the assumption that if the ratio of gage length to square root of the cross-sectional area is kept constant, the elongation for any given material will be constant. Furthermore, in developing this equation he varies the ratio $\frac{L}{\sqrt{A}}$ by varying the gage length, L, and allowing the cross-sectional area, A, to remain constant for any particular material. The equation then might also be considered as having the following form:

$$e = K'' L^n$$

where, $K'' = \frac{K'}{(\sqrt{A})^n}$ of Eq. 2.

In the case of the present investigation a fixed gage length of 2 in. has been used, so Eq. 2 becomes

$$e = K''' A^{-0.5n}$$

and letting $q = -0.5n$ we obtain the formula:

$$e = K''' A^q$$

which is the same as Eq. 1.

That the elongation will remain constant, or nearly so, with considerable variation in the shape of the cross-section of the specimen provided the area is constant, has been pointed out by Martens(4) and Unwin(6); but their tests covered an appreciably smaller range of width to thickness values. In order to further verify this conclusion, some additional tests were made using specimens as shown in Fig. 11. These tests gave results as shown in Table II. The close agreement noted in the elongation values obtained would appear to verify the conclusion given above.

EFFECTS OF RADIUS OF FILLETS AND WIDTH OF ENDS

For this portion of the investigation, it was decided to use only one thickness of material, appreciating nevertheless that the effects of radius of fillets and width of ends might become more pronounced for tests of thinner material. Experience had shown, however, that any such change in magnitude of the effects with decrease in thickness would be so masked by other factors, such as difficulties of properly preparing and testing the specimens, as to preclude the justification of more extensive tests until at least a preliminary series of tests had been made. The details of the various specimens used are shown in Fig. 3, and the test results obtained are given in Table III.

Effect on Tensile Strength and Yield Point:

The tensile strength and yield point values obtained from these tests are so nearly constant throughout for any given type of metal, irrespective of radius of fillets or width of ends, that it was considered unnecessary to plot them. This indicates that the radius of fillets and width of ends have little if any effect on the tensile strength and yield point values within the limits of the variations in the dimensions and for the size of the reduced section used.

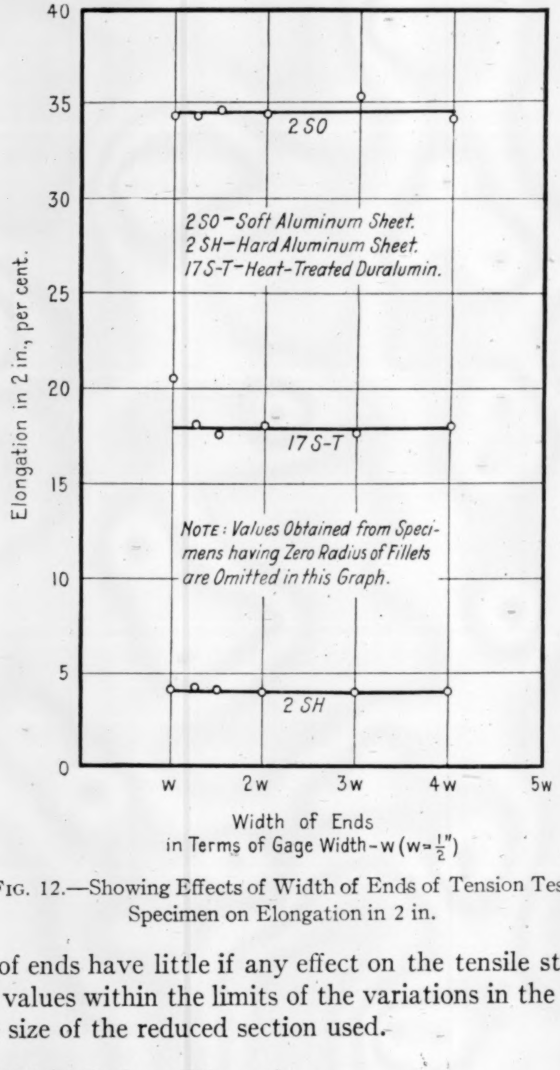


FIG. 12.—Showing Effects of Width of Ends of Tension Test Specimen on Elongation in 2 in.

Effect of Width of Ends on Elongation:

The effects of width of ends of tensile specimen on the elongation in 2 in. is shown by Fig. 12, wherein the elongation values for the three types of sheet metal are plotted irrespective of radii of fillets. In this graph, however, the elongation values obtained from the tests of the specimens having zero radius of fillets were omitted because all of the 2SO and 17S-T specimens broke outside of the gage lengths. It is notable also, that only about 20 per cent of the 2SH specimens having zero radius of fillets broke outside of the middle third of the gage length. These data indicate that the width of ends of tensile specimen, within the range of *w* and 4*w*, has little if any effect on the elongation values of the materials tested.

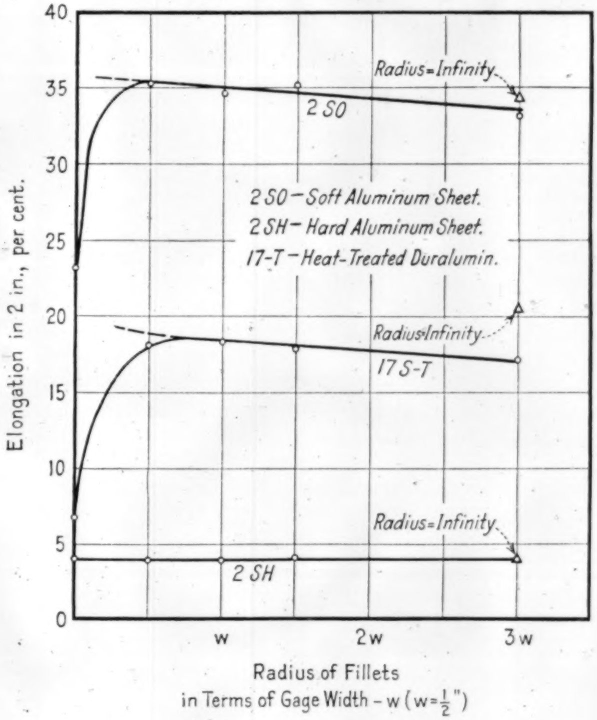


FIG. 13.—Showing Relation Between Elongation and Radius of Fillets.

Effect of Radius of Fillets on Elongation:

The elongation values for the different types of sheet metal have been plotted against radius of fillets as shown in Fig. 13. The erratic values of elongation for zero radius of fillets in the case of the 2SO and 17S-T metals can be explained by the facts that all these specimens broke outside of the gage lengths. In the case of the 2SH metal, as indicated previously, but comparatively few such breaks occurred, and such as did were omitted from the average. The elongation values for specimens with no enlarged ends, that is, infinite radius of fillets, have been plotted opposite the values for radius of fillets equal to $3w$.

The elongation curve for the 2SH metal would seem to clearly indicate that variations in the radius of fillets have little if any effect on the elongation values. This however, does not appear to be true in the case of the 2SO and 17S-T metals. Both these metals show a slight decrease in elongation with increase in radius of fillets from $w/2$ to $3w$. When the radius of fillets becomes infinite, however, there appears to be a slight increase in the elongation. The explanation for the peculiarities shown by the elongation curves for the 2SO and 17S-T metals is suggested, perhaps, by Coker's (3) experiments with reference to variations in stress intensities with changes in contour of tension test specimens. That is, as the radius of fillets is decreased, more of the reduced section undergoes pure tension during testing. One would expect slightly higher elongation values under such conditions. The magnitude of the effect of the radius of fillets, however, is seen to be comparatively small and in the case of the 2SH metal, even though present, is less than the errors of the elongation measurement.

In the case of specimens with infinite radius, it should be noted that the length of the " reduced section " in these specimens would be appreciably more than 2.25 in., with the result that shoulder effects are absent, thus tending to permit of higher elongation values in such types of sheet metals. This is probably the explanation for the higher elongation values for the specimens of 2SO and 17S-T with infinite radius.

CONCLUSIONS

From the results obtained in this investigation it may be concluded that the dimensions of a tension test specimen for sheet metals from 0.005 to 0.250 in. thick may vary between considerable limits without affecting the tensile strength or yield point values.

On the other hand changes in certain dimensions of the test specimen have appreciable effects on the elongation values when measured on a gage length of 2 in. Within the limits of the investigation, by far the major effects on the elongation values appear to be due to variations in the total cross-sectional area of the reduced section of the specimen and follow the law expressed by the equation $e_2 = KA^n$. That is, no one size and shape of tention test specimen having a fixed width and gage length, will give directly comparable elongation values for different thicknesses of the same sheet metal. Knowing, however, the elongation value for any particular thickness of a given kind and condition of sheet metal, the elongation for any other thickness of the same material can be obtained by the use of the comparatively simple formula just indicated. Furthermore, in order to compare elongation values of different sheet metals or different conditions of the same metal it is necessary to use tension test specimens having the same cross-sectional areas in their reduced sections.

In general, the results of this investigation do not indicate that any particular size and shape of tension test specimen for sheet metals is superior to any other from the viewpoint of mechanical properties. In choosing a suitable specimen, therefore, the author would be inclined to give major consideration to other factors such as ease of preparation, duplication, handling and testing. Included in these factors is necessarily the requirement that the size and shape of specimen chosen shall be such that the number of breaks outside the gage marks will be a minimum. Experience has shown that the foregoing requirements are met by the size and shape of sheet tension test specimen shown in Fig. 1.

Acknowledgement.—The author wishes to acknowledge the assistance of Mr. E. J. Holcomb who performed the tests and helped prepare the data.

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DISCUSSION

MR. HARVEY A. ANDERSON[1] (*presented in written form*).—This paper, embodying conclusions regarding the best form of tension test specimen for thin sheet metals, represents probably the most extensive study of the effect of variations in dimensions of sheet specimens which has been made to date. The work here reported together with other work done by Mr. Templin has been of great value to the Section, of Committee E-1, on Tests of Thin Sheet Metals in formulating its report.[2] Although Mr. Templin's paper presents results for three types of aluminum alloys only, it has already been found that his conclusions are apparently applicable to certain other materials. **Mr. Anderson.**

Considerable work was done several years ago on the effect of dimensions of tension test specimens on the test results by H. F. Moore.[3] His report includes a survey of the available literature on the subject together with certain additional tests of round specimens and concludes that the ratio of the square root of the cross-sectional area to the gage length has little effect on any of the properties except the elongation. J. H. G. Monypenny[4] also studied the effects of variations in dimensions and concluded that a constant value of elongation could be obtained for round specimens of various sizes of a given material by keeping the ratio of the gage length to the diameter constant.

The formula which Mr. Templin gives in Eq. 1 to express the relation between the percentage of elongation and the cross-sectional area of the reduced portion of the specimen for a given gage length ($e_2 = KA^n$) represents a refinement of earlier formulas and will be of considerable value in making comparisons between results on different sizes of specimens when values for the constants are available for other materials. His suggestion that the exponential power to which the area is raised in the equation is a function of the hardness of the material and is practically uniform for various materials of the same temper is quite significant.

Additional information on this relationship is given by tests made in the Bell Telephone Laboratories on a number of spring-temper phosphor-bronze strips of various thicknesses ranging from

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[1] Bell Telephone Laboratories, Inc., New York City.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 531 (1926).

[3] H. F. Moore, "Tension Tests of Steel with Test Specimens of Various Size and Form," *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part I, p. 403 (1918).

[4] *The Engineer*, London, August 26, 1921, p. 220.

Mr. Anderson.

0.010 to 0.047 in. Computations of the constants from these results give a relationship of $e_2 = 190\ A^{0.5}$. This material had a tensile strength of about 90,000 lb. per sq. in. and an elongation varying from about 1.5 to 7 per cent in 2 in. for specimens of the above thickness range, all of which were ½ in. wide. This material is, of course, considerably higher in tensile strength than any of the aluminum alloys reported by Mr. Templin. Tests on another phosphor-bronze spring-temper material of still higher strength showed corresponding increases in the values of K and n. The latter material showed a variation in elongation for a given thickness from about 5 per cent in 2 in. for a ½-in. wide specimen (commonly used) to over 10 per cent in 2 in. for a 1½-in. wide specimen.[1]

Three factors which are not considered in Mr. Templin's paper and which may have a bearing on the results obtained with different types of specimens are (1) variations in the gage length, (2) variations in the ratio of the length of the reduced section to the gage length greater than 2 to 1, and (3) variations in the speed of testing. The effect of variations in the gage length are discussed in the papers of Moore and Monypenny referred to above. Tests which have been reported recently to the Section on Tests of Thin Sheet Metals by Mr. Crum of The Stanley Works indicate that there is an increase in the percentage of elongation for a given material over a 2-in. gage length if the length of reduced section is made as much as 4 in., but no further increase in the percentage of elongation in 2 in. with further increases in the length of reduced section up to 10 in. Mr. Crum's findings have resulted in the recommendation of a specimen in the proposed Tentative Specifications for Cold-Rolled Strip Steel[2] with a length of reduced section of 4 in. instead of 2¼ in. as shown in the specimen of the Section on Tests of Thin Sheet Metals.[3] It is expected that further work will be done to determine the effect of such increase in the length of reduced section with constant gage length.

Considerable variation in the strength of sheet metals is known to result from variation in the speed of testing. In the case of certain lead alloys, to cite an extreme example, an increase in strength of 25 per cent resulted from increasing the rate of application of load from 0.02 to 0.5 in. per minute per inch of free length of specimen. Similar considerable variations in strength and ductility due to changing the rate of application of load have been reported for zinc, and smaller variations have been found for certain copper alloys.

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[1] Specimen for plate material, Tentative Methods of Tension Testing of Metallic Materials (Serial Designation: E 8 - 25 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 854 (1925).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 660 (1926).

[3] Figure 1(*l*), Report of Section on Tests of Thin Sheet Metals, Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 533 (1926).

Mr. Whittemore.

MR. H. L. WHITTEMORE[1] (*by letter*).—Materials engineers who have to design test specimens or who have to compare the results obtained when specimens of different forms are used will find great assistance in Mr. Templin's investigation of this subject.

Barba's law has been known for years and materials engineers in comparing materials of different thicknesses have tried to make comparative specimens as nearly geometrically similar as possible. This, however, is in most cases impractical for sheet metal.

Mr. Templin's specimens having the lowest width-to-thickness ratio were $\frac{1}{4}$ in. square—a ratio of one. One cannot help wondering how the elongation of these specimens would compare with the elongation of specimens having a circular cross-section of $\frac{1}{4}$-in. diameter. Probably the elongation would be the same or perhaps slightly less for the square specimens because failure starting at some slight defect in the sharp edges might extend across the section more readily than if failure started at a similar defect on a cylindrical surface.

It would help materials engineers if a direct comparison between specimens having square and circular cross-sections had been made; then we could compare the elongation of specimens of the usual cylindrical form with specimens cut from sheet metal having a width to thickness ratio up to 50.

Provided there has been no cold working of the metal, a shape such as a channel section would, presumably, have the same elongation as an equivalent rectangular specimen. Of course, the thickness of the shape must be a constant. If the formula worked out by Mr. Templin is found to apply to shapes it seems logical to suppose that it applies also to a circular tube having a uniform wall thickness; perhaps to a tube having any form of cross-section.

Obviously the application of the formula to specimens which were not tested in this investigation is not warranted until more experimental work has been done.

It is particularly gratifying to have the results of these tests comparatively free from inconsistencies. This is undoubtedly due to the exceptionally good testing technique employed in Mr. Templin's laboratory. It is to be hoped that we shall have a paper describing this technique in detail in order that testing laboratories for materials may benefit by the experience of others. When a large number of tests are required for the control of a manufactured product, the cost of the work is of great importance. The methods of machining and testing specimens of aluminum alloy could without doubt be used for other metals.

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[1] Chief, Engineering Mechanics Section, U. S. Bureau of Standards, Washington, D. C.

Mr. Templin. MR. R. L. TEMPLIN (*author's closure by letter*).—The author wishes to thank Mr. Anderson for the additional references which he has given. The present paper did not include data pertaining to variations in the gage length but additional data is available in the author's files, which show that the same general law expressed by Eq. 1 of the paper holds for different gage lengths when proper values for the constants are taken. The curves showing the variation in the elongation with change in the ratio of the length of reduced section to the gage length are given in Fig. 6 of the paper.

The effect of variations in the speed of testing has been extensively investigated by the author but was not considered as a variable in the problem covered by this paper because of the space limitations imposed.

In reply to Mr. Whittemore's discussion, a test specimen having a reduced section $\frac{1}{4}$ in. square will have an elongation in 2 in. somewhat higher than a $\frac{1}{4}$-in. diameter specimen of circular cross-section with a 2-in. gage length, cut from the same material. If the two specimens had the same total cross-sectional area they would have the same elongation values over a gage length of 2 in. Again a tubular specimen of the same cross-sectional area would have the same elongation if prepared from the same material. We have made numerous tests to verify this point using various grades and tempers of aluminum and its alloys and using specimens ranging in size from those cut from sheet (0.010 by 0.500 in.) up to those cut from plate (1.0 by 1.5 in.), that is, specimens having total cross-sectional areas ranging from 0.005 sq. in. to 1.5 sq. in. In doing so, we have used round, square, rectangular and tubular shaped test specimens. On the basis of these tests, it would seem logical to assume that specimens cut from channels or angular sections would have the same elongation values if of the same cross-sectional area, provided of course, that such specimens could be properly gripped in testing.

PORTLAND CEMENT RESEARCH[1]

By R. H. Bogue[2]

Research on the constitution and the nature of the reactions of the hydraulic cements began long before the development—we cannot call it the discovery—of portland cement. Even if we pass by the cements of the Romans and other early peoples as merely fortuitous mixtures which have stood through the ages only by the kindness of nature in furnishing favorable and uniform climatic conditions, yet we cannot so easily overlook the hydraulic products which resulted from the extensive and purposeful studies conducted by John Smeaton in 1756, Higgins and Bergman in 1780, Collet Descotels in 1813 and Vicat in 1818. The principle of hydraulic cement manufacture, based on the burning at high temperatures of a proper mixture of limestone and clay, was established through the researches of these investigators, and the product was patented as portland cement by Joseph Aspdin in 1824. This name was given because of a resemblance which the product bore to a natural stone obtained in Portland, England.

Since that time many investigations have been carried on which have resulted in the accumulation of a vast amount of data and occasional glimpses of the laws by virtue of which cement is caused to function as cement. For the most part, these investigations have fallen into one of two major classifications: (1) the chemical problems of composition and constitution of the clinker and the reactions of its constituents in setting and hardening, and (2) the engineering and mechanical problems of proportioning and placing of concretes and their strength and durability under various conditions. Each of these two points of attack is indispensable to the complete understanding of cement manufacture and of concrete construction and permanence. Neither one alone can suffice.

Principle of Fundamental Research

A large proportion of the work which has been reported on composition and especially constitution has been empirical, but such investigations as those of LeChatelier, Michaelis, Kühl, Richardson,

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[1] Published by permission of the Director of the Bureau of Standards, U. S. Department of Commerce. Paper No. 4 of the Portland Cement Association Fellowship.

[2] Research Director, Portland Cement Association Fellowship, U. S. Bureau of Standards, Washington, D. C. The Portland Cement Association Fellowship at the Bureau of Standards consists of a staff of physical chemists, physicists and petrographers conducting research on portland cement under the joint support of the Portland Cement Association and the National Bureau of Standards.

Newberry, Rankin, and many others, have shown that it is possible, by appropriate means, to study the internal make-up of a clinker. Their work has indicated that the behavior of the individual constituents determines in turn the behavior of the cement. However, the identity of even the major constituents is not universally agreed upon, and almost no deliberate and well-ordered attempt has been made to determine the identity and the significance of the less abundant constituents. Guesses have been made, but guesses cannot become the bases for confidence or knowledge.

Unfortunately, the pursuit of research on these problems, with the requirements necessary to their satisfactory solution, involves equipment which is not generally available, which must be especially designed for each study, and which is costly. This is one reason why intensive studies of this order have been made in only a few laboratories. The time period required for such investigations and the great patience as well as technical skill necessary to their final solution also serve to deter those who would obtain results in a brief period, or who have not the patience that is required. This may explain why the Geophysical Laboratory at Washington is the only laboratory in which a thorough investigation has been made of the system lime-alumina-silica, the system important above all others to the several branches of the ceramic industries. It may be observed that in this study some six or eight men, chemists and petrographers, were engaged for about seven years, during which time many thousands of different concentrations, heat treatments and microscopical examinations were made. Several types of high-temperature furnaces were required including platinum for temperatures up to 1600° C. and iridium for temperatures up to 2000° C.

Although the conditions imposed on research of this order are severe, I am convinced that further definite progress in our understanding of the nature and the reactions of portland cement can be made in no other manner. Empirical methods may be used which will bring out relationships, and statistical methods may be applied which will indicate "mean variations," but these will not answer the fundamental questions, "What is the cause of these relationships?—What is the reason for these variations?"

It is not especially difficult to ascertain that two cements behave differently in service—that one sets the more rapidly, is the more nearly volume-constant, is the more rapidly hardening, develops the greater strength, or is the more resistant to corrosive solutions. It is even possible, at times, to find a method for retarding or accelerating the set, for improving the volume-constancy, for accelerating the hardening process, for increasing the strength attained, or for lengthening the service when some given cement is made the object of study. But if a different cement is used, if the nature of the raw materials or the manufacturing processes are changed, if the corrosive salts are of a different type, if the gaging water or the soaking water is allowed to change in any respect, if the temperature varies, if any one of a score of conditions is caused or allowed to change, then we may no longer say with assurance that the data obtained under the first set of conditions can apply equally well to these changed conditions. Just such assumptions have been made not infrequently and with conspicuous lack of appreciation of contributing factors. Empirical and statistical data in the past have served a purpose, but they have given us nothing fundamental. They have told us nothing of the course of the reactions by virtue of which a given composition, under a given treatment, yields a product possessing definite properties, nor the change in these reactions to be predicted by an alteration in the composition or the treatment.

SCIENTIFIC AND THE ENGINEERING POINTS OF VIEW

Much has been written about "science for science' sake." Probably all men who have meditated upon natural phenomena, the orderliness of things from astronomical oscillations, through life processes, down to atomic and electronic symmetry, are overwhelmed with the wonder of it all, are filled with awe, and feel to a greater or a less degree the urge of a "divine curiosity" to learn more. Much also has been written about science for industry, for progress, for civilization. Still, we have seen from time to time evidence of a conflict between "pure" science and "applied" science. The professor in his laboratory, sometimes, as he traces the course of an electron, has regarded the engineer as a rather hopeless materialist. The engineer, sometimes, as he surveys the magnificent product of his skill, has impatiently thought the scientist an impractical dreamer. They stand: the scientist and the engineer: the dreamer, the explorer; and the doer, the builder.

But still another motive for research may be added—a motive which unites the scientist and the engineer—"science for humanity's sake." It is true that the principles underlying the behavior of great forces must be discovered by painstaking research before their application may be most effectively developed. But I do not think there is a research man engaged on the most abstruse of problems who does not experience a gleam of hope, behind his passion for the exploration of the unknown, that sometime, somehow, a great service to humanity may not accrue from his labors. Every real engineer on the other hand knows well that his marvelous capacity to do has been made possible very largely by the dreams and patient toil of some idealist.

It is because I share the viewpoint of the scientist that I am convinced of the importance of intensive fundamental research, that we may give to the engineer ever more of the tools of knowledge, that he may build safer and with surer control, that he may build bigger and with greater confidence, that he may build for greater permanence, that his efforts may be of greater ultimate service to humanity.

This point of view explains why we interpret the great and prime requirements of the industries concerned with portland cement from an angle somewhat different from that of the engineer. It is his function to make the most splendid use, mechanically and architecturally, of the materials at his command. It is the function of the scientist to give greater power to the engineer or the architect. Hence the engineer is concerned with standard specifications, with methods of proportioning, with water-cement ratio. His research is directed to the very important matters of strength and uniformity and serviceability of concretes, the solution of which requires great numbers of tests of many mixtures. His efforts are directed to the correlation of laboratory data with field conditions and the interpretation of the former in terms of the latter. He asks what treatment can be applied to correct certain undesirable conditions, to improve uniformity or strength or permanence of concrete. His thoughts are focused mainly on design and control and his questions are answered for the most part in terms of statistical averages.

The question of the chemist, however, is "What is the nature of the reactions which give rise to the effects observed by the engineer? Why does this material behave so, and that material behave so? What is the reason for these things?" He sets himself to answer these questions, and having solved them he is well on the road to a knowledge of the control of these same reactions. The two functions, that of the engineer and that of the scientist, are equally important, and both are absolutely essential to real progress.

I have taken this much time to indicate our attitude towards industrial research because only by an understanding of and sympathy with this attitude is it possible to comprehend properly the significance and ultimate purpose of our method of attack.

RESEARCH REQUIREMENTS IN PORTLAND CEMENT

The basic problem of portland cement research is concerned with the chemical nature of the substance. To determine the exact combinations of the elements which are present in a clinker or in a set cement is a task of such difficulty that no work yet reported is regarded generally of such indisputable quality in technique and surety of interpretation that it has been accorded universal acceptance. Although the composition shows portland cements to contain nearly 90 per cent of certain combinations of lime, silica and alumina, yet even the compounds formed by these three components in clinker are not agreed upon by different investigators. Some maintain that one of the principal constituents is a solid solution of lime in dicalcium silicate, some that it is tricalcium silicate, and still others that it is a ternary compound containing lime, alumina and silica. The alumina has been said to be present as tricalcium aluminate, but this compound is usually found only in traces by the microscopic examination of clinker and perhaps, after all, much of the alumina is present in a different, still unknown, form. The manner of combination of the less abundant constituents, as the iron oxide, magnesia, soda, potash, titania, etc., has hardly been investigated at all.

There is thus opened up for study an enormous field. It has seemed to us that the only adequate means for investigating these problems of constitution, and also the problems of behavior in service, is by the systematic study of the equilibrium conditions and phases in these systems. It is necessary also to examine first the simpler systems of which portland cement is built up.

In practically all of our work we have first sought to study our products and our reactions through the investigation of the simpler systems, and these in a condition of equilibrium, in the belief that with the knowledge so obtained we may be in position to predict the phases and behavior under conditions of greater complexity and of incomplete equilibrium. We regard these two conditions, the simplification of the systems studied and attainment of equilibrium, of such importance that our fundamental studies have been developed altogether upon these criteria.

Perhaps the most outstanding criticism which may be directed to much of the previous work on this subject lies in the attempt to demonstrate the properties of cement (1) without a proper recognition of the complexity of the material, that is, by examining the commercial product rather than the individual constituents, and (2) in the failure to bring about equilibria in the reactions actually studied. When a commercial cement is examined for solubility, for rate of hydration, for adhesive or cohesive strength, for soundness, or for any of a number of properties, the value obtained is at best only the resultant of many factors any one of which may be capable of so affecting the progress of the reactions that comprehensive interpretation of these effects is impossible. But by learning first the behavior of the simple systems we acquire the information which alone can serve as the basis for the more complicated explorations in the systems of greater complexity.

The necessity for working under equilbrium conditions may not be so obvious but it is of equal importance. Thus if a mixture of limestone and silica in the ratio of $2CaO + SiO_2$ were heated slightly, there might result a mixture of $CaCO_3$, CaO, SiO_2, and perhaps $3CaO \cdot 2SiO_2$ or $CaO \cdot SiO_2$. At a somewhat higher temperature the $CaCO_3$ would disappear and other silicates would be formed, the amount varying with the temperature and time of heating. But finally only $2CaO \cdot SiO_2$ would remain. It is obvious that if we were investigating this mixture to determine if a compound of the above composition existed, we would be unable to answer the question until we had carried the process to the equilibrium condition where only the one phase remained. The reasoning in the three-component systems is more involved, but to determine what the products of complete reaction are in each case, it is necessary to bring about the equilibrium condition.

Of perhaps even greater importance is the capacity of generalization which follows only upon the attainment of equilibria. Two of the most fundamental and significant laws of chemical science are concerned with equilibria: the phase rule and the mass law. It is not possible to investigate every conceivable composition, and an application of the phase rule makes this unnecessary. The establishment of fields and of boundary lines between fields by a moderate number of experiments serves to establish the nature of the equilibrium phases of every point within the field. This has been so thoroughly confirmed in all manner of studies during the half century since its discovery that any apparent departure from it would be regarded with certain distrust of experimental accuracy or of interpretation.

In like manner, an application of the mass law explains and accounts for reactions of hydrolysis, of solubility, of crystallization, of rate of reaction. To attempt to examine and interpret any of these reactions without consideration of the mass law can result only in an accumulation of uncorrelated, unclassified, unexplained experimental data upon limited regions. A statistical average so obtained may serve some purposes, but it is uncertain. As an example of some interest, hydrolysis is to a large degree responsible in tempered cements for the alkalinity, the formation of free calcium hydroxide, the formation of the colloidal monocalcium silicate hydrate or other silicate hydrates, and indirectly for the formation, under certain conditions, of sulfo-aluminate or chloraluminate of calcium, and of other crystallization products. Yet many investigations have been directed upon these conditions with no suggestion of hydrolysis, resulting in interpretations utterly incompatible with a sound reasoning based on the mass law. Of course our laws are merely the human expression of the behavior of the forces of Nature, and our observations are both finite and fallible. But it is not expedient for us to pass over lightly such important and well established generalizations.

INVESTIGATIONS UNDER WAY

In our laboratory, further studies have been made on the system lime-alumina-silica in order to verify the field in the region of portland cement and to settle certain points which have been in dispute between European and American investigators. There seems every reason to hope that, insofar as this system is concerned, general agreement will be attained very shortly. In order to bring this about, we are cooperating with our European friends in the belief that, by getting together on disputed matters, we may eventually iron out the difficulties.

We have made a study in the system lime - ferric oxide - silica in the region of portland cement, which already has been published.[1] By this study we learned that the two known binary compounds of lime and iron oxide, monocalcium ferrite and dicalcium ferrite, occur also in this ternary system and that no compounds of iron oxide and silica or of lime, iron oxide and silica are produced in the region of portland cement. The silica combines with a part of the lime to form the dicalcium silicate and the tricalcium silicate. This is further important confirmation for the existence of tricalcium silicate, for some have claimed that the material described as such was, in reality, a ternary compound containing alumina. But, in our study, alumina was absent. It was further observed, by a comparison of the lime ferrites with the iron-containing grains in clinker, that the iron of portland cement is probably present in some other form than as these calcium ferrites.

We are now exploring the system lime - iron oxide - alumina and, although this study is not yet complete and no definite statement can be made, it appears to be a system of much importance in portland cement. Unfortunately the investigation is complicated by several factors which make the research on this system unusually difficult. It is expected however that the solution of this system will solve some of the conspicuous uncertainties which at present exist with reference to the iron and alumina compounds in cement. Similar investigations will be carried on through several other of the systems until the ultimate nature and equilibrium conditions of practically all of the constituents of portland cement are known.

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[1] *Journal*, Am. Chemical Soc., Vol. 48, p. 1261 (1926).

The second line of attack is necessarily to determine the specific influence of each of these compounds or solid solutions upon the several aspects of cement production and utilization. Each compound found to be present in clinker must be prepared in the pure condition and it must be subjected to rigid examination that we may learn the rôle it plays in the product. The effect of varying the ratio of the principal constituents must be determined—the effect both on the economic aspects of manufacture and on the serviceability in use. And in a like manner we must learn the exact and specific influence of each of the lesser constituents—their ability to serve as fluxes to lower the temperature required in burning, their ability to make for more complete combination of the constituent oxides, their ability to influence the rate of setting, the rate of hardening, the strength developed, the resistance to corrosive waters, the permanency.

All of this involves many individual investigations, independent except that the goal of each is the same. In one series we are studying a number of problems concerned with the relation of composition to burning condition and completeness of combination. We have learned, for one arbitrary composition, the influence of magnesia, of iron oxide and of the alkali oxides as fluxes and as accelerators of the reactions of clinker formation. This arbitrary composition approaches that of portland cement, referred to lime, alumina and silica, but the effect of varying this composition through a considerable range must be investigated to establish the ideal basic mixture. The influence of many other of the minor constituents must then be determined, the influence of such materials as titania, manganese oxides, phosphates and sulfates. And even entirely foreign materials should be examined to learn if certain catalytic influences may not result which will be of importance in the manufacture or utilization of cement. Finally, all of these synthetic products must again be studied to learn the influence of the added materials in the processes of setting and hardening, of resistance to corrosion and of permanency.

A number of studies are under way on the various reactions of cements and cement compounds with water and with salt solutions. In order to study these reactions systematically, each of the cement compounds must be prepared in the pure state, the rate of hydrolysis and hydration of each determined under various conditions, and the end-products ascertained, also under various conditions. It is only by such studies that we may hope to learn the nature of the cementing process, and the nature of and reasons for disintegrating influences. If we know just what constitutents are responsilble for cementing action, then we may direct our efforts to increase the yield of these constituents in manufacture to the maximum compatible with economic operation. If we know the manner by which these constituents operate to effect rigidity and strength in concrete, then we may intelligently seek to control the reactions to the end of obtaining greater rigidity and greater strength. If we know the nature of the reactions which bring about solution, or expansive crystallization, or contraction, then we may hope to delay or avert such reactions by bringing the proper chemical forces to bear upon them.

Much of this program already is under way. The rates of hydrolysis of several cement compounds have been studied. The nature of the end-products has been determined. The influence of various degrees of alkalinity has been ascertained. The reactions of several constituents with various salt solutions have been examined. Certain weakening influences have been analyzed. The conditions of formation and stability of the sulfo-aluminates and the chloraluminate of calcium have been studied in detail with respect to their possible formation in concretes.

An apparatus is being developed for the accurate study of the thermal reactions of the setting process, for previous work by other investigators has indicated that the heat of reaction, or the rate of liberation of heat on tempering with water, bears some direct relation to the behavior of the cement in service.

Throughout a great deal of these studies, much depends upon the accurate identification of compounds or other phases after they are produced. A very important part of our work is concerned, therefore, with this matter. Chemical analysis is inadequate to determine the manner of combination of the constituent elements. Optical microscopic methods seem best suited in our work, and much effort has been directed to the improvement of these methods, and to the extending of their field of usefulness. The preparation of thin sections, the measurement of the crystals in a field, the mounting media, the liquids used for the measurement of refractive indices all have been studied intensively. The purification of selenium for use in the preparation of optically clear melts for the determination of high refractive indices also has been accomplished.

The aid of X-ray analysis is being utilized. It is well known that X-ray methods have found most valuable application in metallurgy and the belief is general that their fields of usefulness may be enormously extended. The X-ray patterns for most of the cement compounds have been determined and it is hoped that the progress of reactions may be followed by this means. The system alumina-ferric oxide may be explored by the method of X-ray investigation.

We feel that the application of new and more accurate methods, developed upon a utilization of modern physico-chemical principles cannot fail to give information and throw light upon aspects of the cement problem which would be impossible without such procedure. This has created the necessity of building and developing much equipment which is not available on the market, and of perfecting methods of control which have not been reported in the literature. We have built three furnaces of different types for use in our studies. These are of the platinum resistance type. Two are capable of reaching a temperature of 1625° C. and are controllable to ±1° C. but the charge is very small. The other was designed to be of much greater capacity. It has been used to 1525° C. and is controllable to ±10° C. In this latter furnace, from 1 to 2 lb. of product may be obtained per day. The reactions are brought about in platinum boats in order to avoid contamination with the lining of the furnace. The engineer will appreciate the many difficulties inevitably encountered in the attempt to design and operate furnaces under such high temperatures and rigid control and absence of contaminating conditions as we have prescribed.

In our work on thermal reactions, an adiabatic calorimeter has been built wherein the temperature control is effected by an application of the photo-electric cell. The mechanical operation of this system is very interesting. Any increase in temperature of the setting cement above that of the water bath at once sets up a small electromotive force in a series of thermocouples placed in the two and this causes a mirror on a galvanometer needle, in series with the thermocouples, to be displaced. A beam of light impinging on the mirror is caused, by this displacement, to be reflected into the photo-electric cell. The cell then allows an electric current to pass through it and, by a series of relays, more current is at once thrown into the water bath. An automatic rheostat operated by a shunt-wound motor controls the amount of the current input at all times.

In several of our investigations it is necessary that we have a measure of the free lime present. Accordingly a special study was made to find such a method, applicable to portland cement and in which the easily hydrolyzable silicates and aluminates of lime would not interfere. The results of this study have recently been published.[1]

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[1] *Industrial Engineering Chemistry*, Vol. 18, p. 739 (1926).

An important part of all scientific research is concerned with the systematic digest of the literature on all correlated subjects. In our program, about a hundred periodicals are examined as issued and abstracts made of all papers bearing on our work. About 1400 abstracts are now in our bibliography and translations from the French or German have been made of about 120 important foreign contributions.

SUMMARY

I have tried in this brief period to bring before you a few impressions of our attitude on portland cement research. I have pointed out the reasons why I feel that further distinct progress in an understanding of the nature and behavior of portland cement cannot be made through empirical or statistical studies but only by the investigation of the fundamental characteristics and reactions of the constituent compounds. I have tried to show wherein the point of view of the research chemist or physicist is necessarily different from that of the engineer. To the engineer is given the great task of erecting lasting monuments, symbolic of his art, but to the scientist must be given the equally important rôle of gazing into the very heart of natural processes, of finding out how the atoms work, that he may by this knowledge give ever greater power to the engineer. I have pointed out the difficulties of this type of research, and have stressed the necessity of building up our knowledge always through the attack of the simpler systems and the attainment of equilibrium in order that we may obtain an adequate foundation to serve for the interpretation of the more complicated systems and conditions of incomplete equilibrium. Finally, I have enumerated some of the more important investigations which are at present under way in our laboratory, some of the unusual equipment we have made use of in the development of our technical methodology, and a few of the results which we have obtained.

Upon the completion of various parts of this program there will be recommended such tests for the practical application of our findings as may be necessary to demonstrate their serviceability, both for efficient production and for effective utilization.

It is my earnest wish that the engineer examine our program, scrutinize our method of attack and assist us as well as himself by a sympathetic cooperation to the end that we may come to a fuller appreciation of the possibilities and the limitations in the manufacture and the utilization of portland cement.

COOPERATIVE CHECKING OF CEMENT TESTING

By S. R. Mitchell[1]

Synopsis

The object of this paper is to promote more reliable cement testing in every phase. To obtain this end, the author strongly recommends from personal experience more cooperative checking of cement testing among laboratories in the same locality.

Representatives from laboratories A and B should visit laboratory C, and all work together on a divided sample of cement, including tests on normal consistency, setting time, soundness, mortar tensile and compression tests and other important determinations. Discussion of specifications and agreements on interpretations will afford a common basis for testing work for the three laboratories. Friendly criticism of technique will eliminate the gross differences due to the personal equation and establish uniform procedure for the remaining tests on the same divided sample of cement.

The next step should be a retest by all three parties at laboratory C, using the revised procedure. Following this work, a sample of the same cement should be taken by each operator to his own laboratory for testing by the revised method. This will afford a check on conditions at each place, such as temperature, humidity, storage, etc. If enough tension and compression test specimens are made up at each laboratory to exchange all around for breaking, then a check will also be obtained on the testing machines.

After participation in a number of cooperative cement tests, the consensus of opinion of the laboratory representatives is in favor of this method for the settlement of differences.

Producers, dealers and consumers represented by mill laboratories, commercial, Federal, state, county, and city testing laboratories who have tried it, find cooperative checking of cement testing a very successful basis for the maintenance of satisfactory business relations and research work.

It is a well-recognized fact that cement testing methods produce very erratic results in the hands of testers who are not experienced nor exceptionally careful. Further, there is probably not one cement testing laboratory in operation where A.S.T.M. specifications are followed to the letter. Many laboratories endeavor to follow some set of standard specifications, but even in these cases the workers are handicapped by wrong conditions for following these standards on account of improper control of temperature and humidity, poor apparatus, lack of time, personal equation, and misinterpretation of the spirit of the specifications. There is available to all cement testers much literature covering the proper procedures for comparatively accurate work using our present methods, but these testers often adopt methods of their own, involving short cuts which may or may not be conducive to accuracy.

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[1] Chief Chemist, Riverside Portland Cement Co., Riverside, Calif.

In some cases, it is true, there are certain operations in the specifications which, under certain conditions, are difficult and commercially not feasible to follow to the letter. An example of the above is where definite weights of cement and standard sand are specified for batches of paste and mortar for soundness and for mortar strength tests. Cases of this nature may be satisfactorily handled by observance of the spirit of the specifications, rather than the absolute letter.

Producers and consumers have agreed upon the standard methods for testing cement, and it is necessary that the same interpretation and procedure be adopted by both parties to maintain satisfactory business relations. This agreement on interpretation and procedure, in order to follow the letter and spirit of the specifications, must come only after cooperative laboratory work involving all parties concerned. That which applies to cement testing will probably apply to the testing of many other engineering materials.

Faulty Apparatus.—In the experience of the author with checking work, four laboratories were found to be working with faulty apparatus, including distorted and worn briquet and cylinder molds, worn tension test clips, worn and tight tension test clip rollers, corroded galvanized iron storage tanks, with 2 in. of sludge submerging the specimens, balances with mixed weights of avoirdupois and metric system, non-sensitive balances, and unevenly worn spots on the bearing blocks of the compression testing machine. Renovation and renewal of the apparatus in these four laboratories is not only responsible for better results, but is stimulating more interest and pride in the cement testing carried out at the laboratories.

Effect of Turning Briquet Molds.—Two laboratories were consistently differing about twenty per cent in the seven-day standard mortar tensile strength tests on the same sample of cement. Cooperative checking of the testing with subsequent investigation of the method step by step taught one of the laboratories that the specifications required turning over of the loaded mold, rethumbing and finishing off. The result of this investigation was the elimination of the differences.

Effect of Abnormal Consistency.—Long times of setting of 4 to 5 hours for initial set and 10 hours for final set, and low mortar tensile strengths were reported by two laboratories. The subsequent checking work on normal consistency reduced the time of set 25 per cent and increased the tensile strength 20 per cent by reducing the water content 0.50 per cent.

Necessity for Checking Sieves.—A difference of 5 per cent in fineness passing the No. 200 sieve between two parties led to the revelation that one was checking the sieves practically every day with samples standardized by the U. S. Bureau of Standards, while the other was checking the sieves only twice each year. Needless to say, a closer check was kept on the sieves.

Errors in Chemical Analysis.—Disagreements in chemical analyses of cement have been clarified by cooperative checking. In one case the wrong permanganate factor for lime was used, while in another the high loss on ignition of 4.25 per cent was traced to moisture absorbed due to poor handling of samples in transit.

Factors Affecting Soundness Test.—Soundness variations between three laboratories were practically eliminated by cooperation. Size and type of boiler, temperature of boiler between 98 and 100° C. and length of steaming period between 4 and 8 hours did not affect the results of the test so much as the temperature of the mixing water and of the cement, the temperature and humidity of the moist closet, the length of curing period in the moist closet before placing the pats into the boiler, the thickness and the amount of troweling of the pats.

Tension and Compression Testing.—Tension and compression cooperative tests by three laboratories failing to agree resulted in checks well within 10 per cent. To accomplish this, irregularities in procedures with respect to the oiling of briquet plates and molds were investigated. Uniform oiling was adopted to avoid oil spots in the mortar, which may cause erratic early strengths due to uneven hydration. Digression from the specifications was found desirable with reference to the amount of mortar per batch to favor the use of three-gang 2 by 4-in. cylinder molds. Each operation was timed. Tamping was systematized also somewhat by agreement on a 1.5-in. loose drop of the tamper.

Many other instances may be cited showing the benefit of cooperative cement testing to all parties concerned.

CONCRETE SPECIFICATION PROBLEMS

By Cloyd M. Chapman[1]

Synopsis

This paper is a general discussion of some of the many problems confronting the concrete-specification writer. The topics discussed include:

1. The subjects to be covered in a comprehensive specification.
2. The necessity for separate specifications for concrete for different types of service.
3. The possibility of developing special cements for particular services.
4. The necessity for the development of test methods for determining the suitability of fine and coarse aggregates and water for making concrete for different uses.
5. The various methods for designing concrete mixtures.
6. The adaptation of present specifications of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete to include the use of the water-cement ratio method for design and for control.

Committee C-9 on Concrete and Concrete Aggregates is charged with the duty of preparing specifications and methods of test, not alone for the various materials, except cement, which may go to make up concrete, but for the concrete itself, as it exists, cured, in the structure.

Sub-Committee VI on Specifications for Concrete of Committee C-9 is engaged in the preparation of Specifications for Concrete. Under the chairmanship of Mr. Sanford E. Thompson this sub-committee has endeavored to bring together the mass of data available and the numerous theories for design of concrete mixtures.

In these days of rapid strides in our knowledge of concrete it has been quite impossible for the man on the job to keep up with the advances made. The result has been, and still is, that at no time in recent years has there been any crystallized general agreement as to how concrete should be designed or specified or fabricated.

Since this committee was organized there have passed in review, some with much fanfare of trumpets and many shouting followers,

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such proposed methods as these, all intended to produce the best of concrete:

1. The void-filling method, based on the use of sufficient cement to fill the voids in the sand and sufficient mortar to fill the voids in the coarse aggregate.
2. The use of fine aggregates giving maximum density, as indicated by maximum density curves.
3. The fineness modulus.
4. The surface area.
5. The cement-space and mortar-void ratios.
6. Predetermination of proportions by strength tests, as embodied in the report of the joint committee.
7. The water-cement ratio.

Each of these methods, and others not mentioned, have been used to a greater or lesser degree in concrete construction work. It appears that the time has arrived when by careful consideration of the merits and defects of these we may arrive at a specification in which merit will predominate to a greater degree than has been attained in the past.

To classify and clarify the problems before this sub-committee, there is presented herewith an outline of what a specification for concrete should cover and arguments commonly offered both for and against the various clauses which go to make up a complete and workable specification.

While this paper does not constitute a committee report, the author has had the benefit of many suggestions and criticisms from the members of Sub-Committee VI on Specifications for Concrete of Committee C-9.

A complete specification for concrete will consist of several sections covering more or less distinct and separate matters. These section headings and their contents might follow some such outline as this:

General.—Covering scope, instructions, responsibilities, definitions, penalties, measurements and kindred subjects.

Materials.—Covering kind and quality, and methods for determining fitness.

Proportioning.—Covering proportions to be used or methods for determining and controlling proportions of materials to be used, the allowable variations in proportions and methods for checking proportions on the job, and for determining the quality of the resulting concrete.

Fabrication.—Covering methods for measuring the batches, mixing, transporting and placing.

After-treatment.—Covering control of moisture and temperature conditions from the time the concrete is in its final position till it has hardened and the forms are stripped.

Let us consider the subject matter of each of these sub-divisions in the order named. In the presentation of this discussion it is assumed that the Specifications for Concrete and Reinforced Concrete given in the Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete[1] are the best and most generally accepted specifications at the present time. It is the purpose of this discussion to point out wherein those specifications may be amplified, modified or improved without altering their scope, arrangement and basic principles more than is necessary to bring them in line with the latest developments in the science of making concrete. In order that we may not have before us too much for consideration at one time, this discussion is limited to those portions of the specifications which deal with the proportioning and preparation of the concrete up to the time it leaves the mixer. Matters relating to placing and after-treatment are omitted.

General:

First, there should be a concise statement of the scope of the specification. To cover the wide range of varieties of service and exposure, a complete and all-embracing specification would be complicated and cumbersome. It would have to include such kinds of concrete as plain, reinforced, rubble, cyclopean, and mere space-filling concrete. It would cover such a variety of uses as footings, walls, floor slabs, beams, columns, fireproofing, sea walls, piles, conduit, roads, walks, tanks, and chimneys. It would have to insure proper concrete to withstand successfully external and internal forces, temperature changes, and moisture changes. Some must be impermeable. Some must withstand chemical solution; some is merely required to fill space; some is used as soundproofing; some requires heat insulating qualities; while with some the ability to withstand abrasion or erosion is of paramount importance. Then again the differences due to kind and character of cement used introduces another variable. Portland, natural, and high-alumina cements are available. Each has its field of usefulness. Since portland cement has by far the greatest field, and as it is the only one Committee C-9 is at present prepared to deal with properly, this discussion will be arbitrarily limited to portland-cement concrete having a required strength in compression, shear and tension and sufficient impermeability to withstand weather exposure and frost action. The statement of scope should make this plain.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part I, p. 303 (1924).

Second, under the general heading, may come instructions regarding the use and application of the specifications. Attention should be called to any alternative either of methods or of materials permitted therein, or any quantities, proportions, strength values or grades which are to be filled in by the engineer to make the specification complete, just as is now done in the Joint Committee specifications.

In this section may also be defined, or reference given to, the authority and the responsibility of the architect, the engineer, the inspector and the contractor. The tendency of late years has been rapidly toward making the architect or the engineer responsible for methods to be used for securing the desired qualities in the concrete, while the contractor is held responsible for securing those qualities. It is then the duty of the inspector to see that the engineer's methods are carried out and that the contractor's product meets the requirements of the specifications.

The rules governing supervision and inspection may well be set forth in detail in a separate part of the contract, or in an appendix, rather than form a part of the specification for concrete, but reference to such rules should appear in this section of the specification.

Definitions of terms used in the specifications should be given in the general section whenever these terms are used with any special or limited meaning, other than the common or dictionary meaning. Terms which are defined by the Society or appear in other standards of the Society should be used with similar meanings in all new specifications. If not so used attention should be called to the difference.

Penalties to be imposed for non-compliance or for faulty compliance with the specifications may be set forth under this general heading or they may be referred to here and be set forth in full in another portion of the general contract. This term "penalties" does not refer to rejection of materials or to use of objectionable methods, as these matters are covered in detail in subsequent sections of these specifications. The penalties referred to here are those for failure to attain the end desired, such as strength, impermeability, appearance, etc. They may range all the way from tearing out the work and replacing it to the application of a coating to improve appearance.

Methods of measurement, both of the volume of the concrete and of the area of the form surface may be specified in this section or reference made to them. This is a matter which is often subject to dispute between concrete contractor or form contractor and the architect or engineer. Definite rules should be established.

Materials:

We may next consider the materials to be used, their kind and quality. First the cement. There is opportunity here for either portland or high-alumina or natural cement to be included. Since the assumption is made earlier in this paper that we are to consider only portland-cement concrete, we may at once dismiss the other two kinds from the discussion. It should be borne in mind, however, that these other cements are in use and that specifications covering the concrete made with them are a part of the problem before us. While the limits of this paper will not permit of their inclusion, they may not thus easily be put aside. In due time they must have proper consideration.

The American Standard Specifications for Portland Cement are generally recognized and as they are the standard of this Society they will, of course, be adopted and referred to in any standard specifications for portland-cement concrete adopted.

The present portland cement specifications are, however, considered by many as far from perfect, and subject to considerable improvement. These improvements will probably come about gradually and they may be hastened by a more general recognition of the weak points and shortcomings of the present specifications.

Several suggestions have been made looking to improvements, and while it is not within the province of Committee C-9 to change the cement specifications, it is its privilege and duty to pass on to the committee on cement such proposals as may affect specifications for concrete. Among these proposed changes in portland cement specifications are higher strength requirements, a greater degree of fineness, a more rapid hardening, a more reliable strength test, a short-time strength test, a truly indicative soundness test. Portland cement manufacturers are awake to the desirability of these improvements and some are now taking active steps to accomplish them, at least in part. Cements are now being produced by several manufacturers which are considerably in excess of the standard strength and fineness requirements. The more generally the matter of improvement is discussed, the sooner we are likely to see advances made. What the user demands the industry will supply—at a price. The answer to the request for improved cement is the counter query: What price cement?

Perhaps the economic answer to the steadily increasing and insistent demand for a cement better adapted to certain special purposes is the production of new kinds of cement. Concrete road making alone consumes enough cement to justify carefully considering the possibility of developing a new cement possessing to the highest attainable degree those qualities most important in this class of service. Instead of trying to incorporate in a single cement all the qualities demanded by all classes of service, might it not be cheaper and better to vary the cement to suit the service?

Sand is the next most important ingredient in concrete. In fact, it would seem to some the most important, since it is probably true that in so far as quality of materials is concerned, it is the sand which causes more trouble than any other component in concrete. By that is meant that when trouble occurs on construction work, when the concrete does not set up, or any unusual or unaccountable thing happens on the job and the "trouble shooter" or expert is sent for, it is the sand that receives his first and most careful attention, and more often than not, it is herein that the fault lies.

Whatever the method used in designing concrete mixes or of specifying proportions, it is necessary that the sand be suitable for the purpose. Regardless of how much leeway is given in the matter of mechanical analysis or fineness modulus, it is still necessary that the grains be clean and uncoated, that they be sufficiently hard, that the organic content be not in excess, and that it run sufficiently uniform in quality so that proportions once established may not have to be frequently or radically altered to maintain high quality in the concrete. These qualities are essential. It is also highly desirable that the sand be well graded, that the larger particles predominate. This is largely an economic factor. A well graded aggregate may be used in greater quantity per unit of cement than a very fine aggregate. This is true whether the proportions used be determined by fineness modulus, surface area, water-cement ratio, or other means. A fine sand requires more cement than a coarse graded sand, to give equal strength in equally workable mixes.

It should also be free from objectionable foreign material, that is, material not sand, such as lumps of clay, ice, etc. To insure acceptable quality in the sand, many tests have been used and many more proposed. Some of these tests have been made standard and others of equal or greater importance have not. For instance, while we have a standard test for organic impurities, we have none for hardness, or for presence of coating on the grains.

Consider hardness. It is all very well for the specifications to state, as most of them do, that "the sand shall consist of clean, hard, strong, etc., grains." How is one to distinguish a sand having grains just hard enough to be accepted from another sand having grains just soft enough to be rejected. Imagine specifications for cement which required that briquets made with the cement be "strong and hard," and which said nothing more about either strength or hardness. That is closely analogous to the present state of the commonly used specifications for sand in so far as they relate to strength and hardness.

It is true that in localities where the natural sands are siliceous it makes but little difference,—all the sands are hard and strong. But there are localities having sands quite unsuited to use in high grade concrete because of their softness and weakness. On what basis are they to be rejected? How is their hardness, or lack of it, to be determined? We should either have a test for hardness or omit the word from our specifications.

Much the same may be said about cleanliness. When is a sand unclean? Shall we continue to specify that sand shall be clean when we provide no way of determining when it is unclean?

Then there is the matter of soundness. Some concrete must resist repeated alternate freezing and thawing year after year. To do so requires that the aggregate—both the fine and the coarse—shall be able also to resist this exposure without crumbling. How shall the sand's ability to do so be proved before it is placed in the structure? It has been suggested that one or more of the following tests be used as a measure of soundness:

1. *An absorption test.* It is claimed that a sand having a high absorption capacity will not endure oft-repeated frost action, and that therefore an absorption test may be used as a measure of soundness.
2. *Sodium sulfate test.* This test, involving alternate saturation with sodium sulfate solution and drying out, is advocated by some and by others condemned as too severe, and as not having any direct relation to soundness.
3. *Sodium chloride test.* This test resembles the sodium sulfate test, but is less severe.
4. *Freezing and thawing.* This test is an attempt to reproduce natural conditions, but it is difficult and expensive to make and involves expensive equipment.

Out of these tests, or some other, should be developed a reasonably reliable method for determining soundness and for rejecting unsound sand.

Of the other characteristics of sand usually included in specifications, there is an admitted or implied vagueness or uncertainty as to what is permissible, which puts the decision up to the engineer or the architect. It is commonly stated that sand shall be free from "injurious amounts" of certain objectionable constituents such as dust, lumps, soft or flaky particles, etc. This leaves the question of what constitutes an injurious amount to be determined by tests of the resulting concrete or to the decision of the engineer.

Perhaps it is better to leave these matters in a somewhat indefinite shape rather than to definitely demand certain qualities for which there is no standard method for determination.

Regarding sand tests in general, it is commonly assumed that if a given sand gives test results equal to Standard Ottawa Sand, it is a satisfactory sand for concrete. Many present specifications base the acceptance of the sand on such a comparison with Standard Ottawa Sand. There are, however, occasional cases in which such a comparison is not only useless but very misleading. Many natural sands which show relatively low test strengths in mortar mixtures, will, when combined with suitable coarse aggregate, make entirely acceptable concrete. To accept or reject such a sand, therefore, solely upon a determination of its strength ratio to Ottawa sand, would be quite unsatisfactory. Such strength comparisons are, however, indicative, though not conclusive, and they will probably continue to be used by some engineers while other and better methods are gradually forcing it into the background.

An improvement on the Ottawa sand comparison test is offered in the direct determination of the strength of concrete made with the sand under investigation. Such tests should be made using all of the materials which are to be used on the job and mixing them in the same proportions that will be used in the work. Such a test also affords an opportunity for determining the proportions of aggregate which may be used with a given water-cement ratio, and also the strength which may be expected to result therefrom.

Proceeding now to the quality of the coarse aggregate, many of the remarks made above apply just as much to coarse aggregate as they do to sand. Grading is relatively unimportant. Maximum and minimum size limits are desirable and are customary. Within those limits, size has but small effect on quality. The larger the coarse aggregate is, the greater the tendency to segregate, and the less workable is the concrete, if other factors remain the same.

After size has been specified there remains no other quality for which standard methods of test have been adopted. It is customary to add a list of attributes with no method of determining them. This list is usually followed by a list of things which must not be present in "injurious amounts" but with no guide as to what constitutes an "injurious amount."

For some purposes it is of very vital importance that the coarse aggregate used in the concrete shall have certain characteristics. For pavements subjected to iron wheel traffic the coarse aggregate must be tough. A strong, hard, but at the same time brittle, aggregate is unsatisfactory. In the matter of soundness, it is probably more important that the coarse aggregate be sound than that the fine aggregate should be.

It is true that many attempts have been made to develop methods of test for coarse aggregate which would evaluate several of its desirable qualities. Tests for toughness of stone have been developed to a point where they find application in some classes of work. But for concrete aggregate no standard for toughness or method of determining it is in vogue.

Wear tests, made in any one of several forms of apparatus in which the sample is continuously turned over in a closed rotating container either with or without shot or other bodies present, have been tried out by many. Some variations of this test are in use by several highway departments. Some form of rattler or Deval machine is used. Sometimes the aggregate is put into the machine alone and sometimes iron shot of various sizes are included in the charge. This test does not give a value for any one quality of the aggregate. What it determines is really a sort of composite of brittleness, hardness, friability, angularity and cleavage.

If a sample of water-worn gravel is tested in comparison with a crushed rock of exactly the same quality and kind of stone, the rounded gravel shows a far higher coefficient of wear than the angular, sharp-edged crushed stone on account of the ease with which these corners and edges wear off under the action of the test.

Just what relation exists, if any, between the results an aggregate gives when subjected to this type of test, and the quality of the concrete it will make has not been sufficiently demonstrated to establish the test as a necessary or desirable one upon which to base acceptance or rejection of concrete aggregate, except as mentioned above in connection with highway work. Much remains to be done along these or similar lines, to develop some form of test which will properly classify a doubtful aggregate.

Water should, of course, not contain too much of a substance which will be injurious to the concrete but just how much of an objectionable substance will be injurious is unknown. At present it is customary to make a general blanket statement of prohibition, with no method of distinguishing between acceptable and rejectable water. If there is a possibility of water being used which will injure concrete there should be tests upon which it may be rejected. The present usual form is but an open admission that little or nothing is known of the action of impurities in the water.

Proportioning:

The section dealing with proportions of ingredients includes those vital elements of the whole subject which have had such a large amount of attention for the past ten years. Ever since it was first proposed before this Society that we abandon the use of arbitrary proportions, such as 1:2:4 or 1:3:5, and substitute therefor proportions which by previous test had been found to yield concrete of the desired quality, this section of concrete specifications has been undergoing many changes.

To properly cover this matter requires a complete knowledge of the influence of the various factors which affect the quality of concrete. Many factors have been investigated, and from time to time put forward as the ruling factor controlling one or another quality of concrete.

Feret many years ago maintained that the best concrete would result if the aggregate were made up of particles having a size relation which would give maximum density. He worked out granularmetric composites of aggregates which would meet this requirement. These curves were the familiar triangular diagrams using three coordinates. His theory was generally accepted as sound but it was often found impractical and uneconomical to control the gradation of the aggregate. This would usually involve screening the aggregate into a series of sizes and recombining in the proper proportion.

In this country Fuller and Thompson worked out a method of securing high-grade concrete which was based on Feret's density principle, but which also took into account the cement in the mixture, utilizing the curve of mechanical analysis for convenience in studying the grading.

For many years investigations centered around studies of the effect of the grading of the aggregate upon the quality of the concrete. The answer was elusive. There seemed to be a relation between grading of the aggregate and strength, but it was extremely difficult to determine just what that relation was. Then Abrams came forward with the proposal that it was not maximum density necessarily, but a certain kind of relation between sizes of particles which might be expressed as a factor or modulus. This fineness modulus, used in conjunction with the water-cement ratio, was offered as a means of designing concrete of a given strength or of predicting what the strength of a given concrete would be. This proposal seemed to possess merit but some investigators got results which cast some doubt upon its universal application.

Edwards then suggested that it was the variation in the total surface area of the particles of an aggregate which primarily governed the strength of concrete. He worked out a method for determining (or approximating) the total surface area of a given sample of aggregate. Mixes were to be designed on this basis. Investigation of this method also showed that there must be some other factor having such an overtowering influence on the quality of concrete that at times the surface area of the aggregate is of minor importance.

Talbot then proposed that concrete design be based on the ratio of the voids in the aggregate to the cement when basic water content is used. This method also was found to possess merit, but after many tests each and all of these methods have been found wanting under some conditions.

The most important proposal is that first advanced in 1918 by the Research Laboratories of the Portland Cement Association at Lewis Institute. It is based on the extensive investigations of Abrams and his associates of the relation between the ratio of the total water content to the cement content of the fresh concrete and its final strength. Those investigations show, and field tests have in general verified, that within the usual limits imposed by workability and with suitable aggregate, the strength of concrete is a function of the ratio of the volume of the water put into it to the volume of cement used. With the water-cement ratio kept constant, the quantity and grading of the aggregate added (within the limits mentioned) will not materially affect the strength of the resultant concrete.

This proposal has met with more general acceptance as a workable basis for concrete design and means of field control than any of its predecessors. In fact, its forerunners are looked upon as but indirect indicators of the water-cement ratio likely to be used with a given sand. For example, a sand having a large surface area factor produces relatively weak concrete because more water is required to wet the sand and cement and make a plastic mix than is required with a sand having a small surface area factor. Or a fine aggregate having a low fineness modulus makes weaker concrete than a coarser sand with a high fineness modulus because of the higher water ratio (and consequently lessened density) necessary to make a workable concrete. Similarly the cement-voids ratio method is an indirect measure of the water-cement ratio required to make a plastic mix.

We now have in the water-cement ratio another and very effective instrument for use in designing concrete mixes and especially for use in controlling the quality of the concrete on the job. With aggregate of known and tested quality and suitability it is possible to predetermine and specify the water-cement ratio which will give concrete of a quality not lower than that required by the design. Where the magnitude of the operation justifies the time and expense incurred, preliminary tests of concrete, made with the same materials that are to be used on the job and mixed in the same proportions, are advocated.

These tests should be made with the same cement, fine and coarse aggregate and water and mixed in exactly the same proportions, allowing for moisture in the aggregate, and preferably mixed in a concrete mixer just as they will later be handled on the work. Having made such test runs it may be expected that with proper precautions against any increase in the water-cement ratio and against a radical change in the quality of the aggregates or in its moisture content which may not be allowed for, the quality of the concrete will average as high as the test specimens. In other words, having carefully established by tests the proportions of all materials which will yield the desired quality of concrete it is only necessary on the job to make sure that the water-cement ratio so established is never exceeded and to keep an eye on the reasonable uniformity of the constituent materials. If this is done, the concrete may be expected to be of the quality designed.

The present Joint Committee specifications permit proportions to be fixed either by trial strength tests made in advance of use or by selecting proportions from a table which is based on the fineness modulus of the aggregate. The method outlined above is based on tests made in advance of use and the water-cement ratio method in combination. If this combination method were incorporated in the Joint Committee specifications the change would entail but small alteration in the wording of Sections 27 to 30, inclusive.

As an example of how this introduction of the water-cement ratio into the specifications as a method of both specification and field control, the four sections referred to are here presented in a revised form, as follows:

V. PROPORTIONING AND MIXING CONCRETE

A. *Proportioning*

27. The unit of measure shall be the cubic foot. Ninty-four pounds of cement (one bag or ¼ bbl.) shall be considered as one cubic foot.

28. The method of measurement for all component materials shall be such as to insure uniform proportions in each batch. The aggregate shall be measured separately by volume or weight. In volume measurement the fine aggre-

gate shall be measured in a manner which will take into account the change in volume due to variations in moisture content and will maintain uniform proportions. In volume measurement of coarse aggregate, it shall be measured loose, without compacting.

The water shall be so measured as to insure that the volume indicated in the column giving maximum water content, expressed in gallons of water per bag of cement in Table IV and inclusive of the water in the aggregate, shall at no time be exceeded.

TABLE IV.—PROPORTIONS FOR CONCRETE.[a]

Part of Structure	Water, maximum, U.S. gallons per bag (94 lb.) of cement	Approximate Proportions by Volume			Concrete Strength Assumed as Basis for Design, lb. per sq. in. at 28 days
		Cement, bags of 94 lb.	Fine Aggregate, cu. ft.	Coarse Aggregate, cu. ft.	
Footings					[b]
Beams, Girders, Slabs					
Columns					

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[a] The engineer should determine, by tests, using the same aggregates, cement and water, that will be used on the job, the proportions of aggregate and the water-cement ratios necessary to produce concrete of the required qualities. Where this is impracticable the water content may be determined by the following table:

Desired Strength of Concrete in Compression at 28 days in pounds per square inch	Water, U.S. gallons per sack of cement 94 lb.
3000	6
2500	6.5
2000	7.25
1500	8

For the approximate proportions of fine and coarse aggregate, Appendix 16 may be used as a guide. Appendix 16 is based on volumes of dry aggregate compacted by rodding in the measure, as specified in the Standard Method of Test for Unit Weight of Aggregate for Concrete (Serial Designation: C 29 – 21) of the American Society for Testing Materials. Corrections should be made in the quantities given in Appendix 16 to take account of the bulking effect of moisture in the fine aggregate. The bulking of fine aggregate (swelling) due to contained moisture, and the method of placing it in the measure, may result in a reduction of 25 per cent in the actual quantity of fine aggregate, as compared with that obtained by dry measurement by the standard method.

[b] Concrete strengths to be used as the basis for design shall be filled in by the engineer.

29. Concrete may be mixed in approximately the proportions indicated in Table IV. The amount of water per bag of cement shall in no batch exceed the quantities given in the table. The proportions of fine aggregate and coarse aggregate may be varied to suit conditions and to give the desired plasticity and workability except that the volume of the coarse aggregate shall be at least equal to, but not more than two times, the volume of the fine aggregate. The amount of coarse aggregate shall not be great enough to cause segregation, harshness in working or to produce a honeycomb concrete.

Variations in the grading of the aggregate which affect the consistency and workability of the mixed concrete may be compensated for by variations in the proportions used, within the limits defined herein.

Variations in the water content of the aggregate shall be compensated for by corresponding variation in the water added to the batch so that in no case shall the specified maximum water content be exceeded.

The engineer shall have the right to make any changes in proportions or materials that may be necessary or desirable, and proper adjustment of compensation will be made in accordance with the provisions of the contract.

In general the proportions of aggregate to cement and water shall be such as to produce concrete of suitable workability which can be puddled readily around reinforcement in the forms. No concrete shall show a slump in excess of the limits specified in Table V unless authorized by the engineer.

TABLE V.—WORKABILITY OF CONCRETE.

Type of Concrete	Maximum Slump, in.
1. Mass concrete	—[a]
2. Reinforced concrete:	
(a) Thin vertical sections and columns	—[a]
(b) Heavy sections	—[a]
(c) Thin confined horizontal sections	—[a]
3. Roads and pavement:	
(a) Hand finished	—[a]
(b) Machine finished	—[a]
4. Mortar for floor finish	—[a]

[a] The engineer must insert the maximum slumps permitted. The slump test requirement is intended to insure concrete mixed with the minimum quantity of water required to produce a plastic mixture. The following table indicates the maximum slump desirable for the various types of concrete, based on average aggregates and proportions:

Type of Concrete	Maximum Slump, in.
1. Mass concrete	3
2. Reinforced concrete:	
(a) Thin vertical sections and columns	6
(b) Heavy sections	3
(c) Thin confined horizontal sections	8
3. Roads and pavement:	
(a) Hand finished	3
(b) Machine finished	1
4. Mortar for floor finish	2

Slump tests shall be made as described in the Tentative Method of Test for Consistency of Portland-Cement Concrete for Pavements or for Pavement Base (Serial Designation: D 138 – 22 T) of the American Society for Testing Materials.

Frequent tests shall be made throughout the work, as directed by the engineer, to determine whether the concrete is of the quality specified and to check the consistency.

These tests shall be made in accordance with Section 4 and at the expense of the owner. The contractor shall provide such facilities as may be practicable for carrying out the tests, and shall cooperate in every way to the end that concrete of desired quality shall be obtained.

B. Consistency of Concrete

(This sub-division of the Joint Committee Specifications may be omitted, as its contents are included in the above.)

It is evident that these specifications, in common with the Joint Committee specifications upon which they are based, require the attainment of only one quality, strength. In some types of service it is more important to have impermeability than high strength. Yet no mention is made of permeability, no limits set for absorption and no means provided for insuring an impermeable product. This is a serious shortcoming of our present specifications, which must have careful attention if we are to produce concrete that we know will be durable as well as strong.

Fabrication:

In considering methods for measuring materials, it is fair to assume that regardless of the type of specification for concrete used, it will be one of the fundamental requirements that the component parts bear a constant quantity relation to each other. To insure such constant proportions it is necessary to consider the means and methods available for measurement. Materials may be measured by weight or by volume. Volume measurements may be made with the materials dry, damp or inundated. We will consider the relative advantages of these methods as they apply to the various constituents of concrete.

In the case of cement, measurement by weight is very much to be preferred. Except when bulk cement is used this matter presents no particular problem since the cement is delivered in packages of uniform weight. When bulk cement is used provision should be made for the accurate weighing of each batch. It is practically impossible, except with methods involving careful and painstaking manipulation, to measure loose cement by volume with an acceptable degree of accuracy. In the laboratory it may be possible to measure cement by volume with such accuracy as might be required in the field but we have no method by which cement may be measured by volume on the job within allowable limits of variation.

The water to be added to a cement mixture presents a vastly simpler problem. It can be measured with about the same degree of accuracy that it can be weighed and that with very simple apparatus. It is the water which enters the mixture as a part of the aggregates—both fine and coarse—that is most difficult to handle. If the water content is to be governed with accuracy it is necessary to know and to allow for the water in the aggregate.

In a 1:2:4 concrete where the aggregate is being accurately weighed, an increase of 2 per cent in the moisture content of the fine aggregate and an increase of 1 per cent in that of the coarse aggregate would result in approximately one gallon of additional water for each bag of cement used, unless correction is made for this additional water. With the inundation method of measuring the fine aggregate, an automatic correction for the moisture in the sand results, since the sand is brought to a uniform condition of moisture, namely, saturation. But the inundation method does not automatically correct for differences in the water content due to variations in the voids in the sand of various batches. An increase of 3 per cent in the voids in the sand being measured, would result in an increase of about $\frac{1}{2}$ gal. of water per bag of cement in a 1:2:4 mix. If at the same time the moisture content of the coarse aggregate increased 1 per cent there would be a total increase of a gallon of water per bag of cement.

With some varieties of impervious materials, such as pure silica, practically all moisture is on the surface of the particles. With more porous materials, including some limestones, a large proportion of the water present is in the pores of the particles. Such absorbed water does not affect the consistency of the resulting mixture, but it acts as a reserve supply which may be drawn upon by the cement to complete the hardening process after surface water has been combined. The question arises then whether aggregates of a vitreous impervious nature should be handled differently from those of a porous absorptive texture in the matter of moisture content.

The measurement of the fine aggregate for concrete presents one of the troublesome problems of the art of making uniform concrete. The variability of the material as regards moisture content, grain size, density, impurities, and in some cases the character of the rock composing the particles, all introduce difficulties which have kept the inventive minds of concrete engineers busy.

Here again there are two methods of measurement available, namely, by weight and by volume. Measurements by volume may be made loose, compacted or inundated. If made loose the presence of water may cause a variation in absolute volume measured of as much as 30 or even 40 per cent. A cubic foot of dry, fine sand, when moistened, may increase in volume to 1.3 or even 1.4 cu. ft. The amount of the increase depends upon the amount of water added and upon the grain size of the sand. A fine sand bulks more than a coarse sand. As successive increments of moisture are added to sand the bulk increases up to a point a little below the saturation point. It then falls rapidly until the saturation point is reached, when its bulk is practically the same as it was when dry. The amount of water which this saturated sand contains is dependent upon another of the variables mentioned above, namely, density, or percentage of voids.

The weight of a given sand measured by volume in a saturated or inundated condition will vary with the density or percentage of voids in the particular batch being measured. The density is dependent partly upon grain size. A coarse, well-graded sand is denser than a uniformly fine sand. With a homogeneous sand, as, for instance, a pure siliceous sand, the voids increase as the density decreases. If the measurement of such a sand is made in a saturated or inundated condition, and then both the sand and the inundating water are used in concrete, there will result two variables. The amount of sand will vary with its density and the amount of water will vary inversely as the density.

While measurement of sand in a saturated state obviates the necessity for correcting for the original moisture content of the sand, it introduces another variable, that of density, which may be as difficult to handle and cause variations of approximately the same magnitude as the variable it tends to eliminate. Perhaps a remedy for this difficulty might lie in the use of a simple and quick method of determining density or voids. One simple way would be to weigh a quantity of the sand in an inundated condition. Knowing the apparent specific gravity of the sand, its density or voids are at once known by reference to a previously prepared table. For instance, if a 200-cc. flask full of unundated sand weighs 410 g. and the sand is known to have a specific gravity of 2.5, then the percentage of voids in that particular sample is 30. Or if the weight were found to be 407 g., the percentage of voids would be 31. This simple method presupposes the uniformity of the specific gravity of the sand and in most cases this assumption would introduce smaller error than the assumption now commonly made that the voids in sand from a given source do not vary.

If the measurement of sand is made by volume with the sand in the condition in which it arrives on the job, then both the bulking effect due to moisture and the variable moisture content should be taken into consideration. This is neither as difficult a problem or as reprehensible a practice as some would have us think, provided proper apparatus is used and calibrated. The still all-too-common wheelbarrow method of measuring aggregates is subject to such enormous variation that, compared with it, suitable well-designed volume measurement mechanisms are a big step forward. While the volume measurement of damp sand is not perfect, yet it is so far in advance of the haphazard wheelbarrow measurement that it should be strongly encouraged.

When closed containers are used in the volume measurement of sand, such as the diamond-shaped measuring hopper with top and bottom cut-off valve, it is possible to measure with a variation of less than 10 per cent, provided the measuring hopper is calibrated for the average moisture content of the sand. Such a variation is not serious, especially when the water-cement ratio is used as the governing factor in securing a specified quality.

If sand, even in a moist condition, is compacted into a suitable container even less variation than 10 per cent may be attained. No practical method for carrying out such a system has been used to any extent. It may, at least for the present, be dismissed from further consideration.

The measurement of coarse aggregate seems to have caused less trouble, and to be a comparatively easy problem. Coarse aggregate generally flows more freely, fills a container more readily, uniformly and completely than sand or other fine aggregate. It is also much less subject to volume changes due to variations in moisture content. In fact, unless a coarse aggregate contains a considerable amount of small particles its volume will be but slightly affected, if at all, by moisture changes. For this reason either volume or weight measurement of the coarse aggregate is satisfactory provided the methods and apparatus used are of the best available types.

Mixing the concrete presents much less subject matter for argument than it did a few years ago. Questions of the type of mixer, the order in which the materials should be introduced into it, the time of mixing, the speed of the mixer, and kindred moot points were much discussed a number of years ago. That discussion led to numerous investigations and tests and as a result there seems to be but little diversity of opinion in these matters.

Occasionally an engineer wants a particular type of mixer and thinks none other as good. Another may want an abnormally long mixing time or may require that the other ingredients be mixed dry before water is added. In general the requirements on mixing appearing in the specifications of the Joint Committee seem satisfactory and present no particular problem. It will not be further discussed here.

The path of the concrete specification writer is not a smooth one. There are many gaps in the present forms to be filled and many vague and uncertain statements to be made specific and clear. The progress made in some of these details during recent years can hardly be described as rapid, while in others very decided advances may be recorded. However, we are in a better position to-day to write a concrete specification that will insure a safe, sound product than we were when the present forms of specification were written. It is in order to proceed with the work and carry it through.

DISCUSSION

Mr. A. T. Goldbeck[1] (*presented in written form*).—Mr. Chapman is to be congratulated on the thoroughness with which he has covered the subject assigned to him. I am glad to hear him state that qualities other than compressive strength should be recognized as the governing ones depending upon the class of structure and the service which that structure will be called upon to render. He has well pointed out that resistance to temperature and moisture changes, to chemical action, to erosion and abrasion, impermeability and low absorption might also be qualities greatly to be desired under particular circumstances; and to these qualities might be added high resistance to cross-bending, high tensile strength, fire resistance and several other properties required under different service conditions.

Mr. Goldbeck.

Very properly Mr. Chapman criticizes the time-honored general clause contained in most specifications for both fine and coarse aggregate to the effect that it shall be clean, hard, durable, free from injurious amounts of dust, soft or flaky particles, etc. It is quite true that these are general terms for which, at the present time, there is no standardized numerical measure and certainly research agencies might well concentrate more effort to the determination of proper means for evaluating these terms. Much thought has been devoted in the past to the formulation of tests for some of these qualities. The problem has been a baffling one, otherwise we should have suitable tests for them at the present time. Specification writers have recognized the inadequacy of such general clauses, but nevertheless it has been thought best to insert them for fear their omission would lead to the attempted use of materials which might be unsuitable even though complying with the more definite portions of the specifications. It is my belief that notwithstanding the admitted inadequacy and general nature of such descriptive terms we had better retain them in our specifications until such time as we can more definitely describe them. Some progress has been made along this line but it will necessarily be slow because of the difficulties attending the problem.

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[1] Director, Bureau of Engineering, National Crushed Stone Association, Washington, D. C.

Mr. Goldbeck. Often some of our more definite tests for fine aggregate are credited with too much importance. How often have we heard of the rejection of sand simply because it had a 95-per-cent strength ratio as compared with Ottawa sand while the specifications required 100 per cent? Technically, such a rejection was proper since the sand did not comply with the specifications, but such specifications should not be written. Tests made in a number of different places have well shown that the strength ratio is a function of the cement as well as of the sand. There is no reason why sand with a lower strength ratio than 100 may not be used so long as the proportions of the concrete are made such as to produce the desired qualities.

With regard to tests for coarse aggregates, much remains to be done. It would be desirable to make a thorough study of proper tests for all aggregates so that they might be specified on a strictly comparable basis whenever this is feasible.

Mr. Chapman has stated that the grading of the aggregate is a relatively unimportant matter. So far as strength is concerned, this probably is so, but grading is very important to the contractor, for the yield of the concrete is very greatly affected thereby. Moreover, the grading of the aggregate is a very important matter to the engineer, for by the use of the proper size and grading of the coarse aggregate he can bring about considerable economy without any sacrifice in strength. For instance, as a typical example, with $\frac{3}{8}$ to 1-in. coarse aggregate, 3000-lb. concrete might cost \$5.95 for the materials per cubic yard of concrete as against \$5.48 when $\frac{3}{8}$ to $2\frac{1}{2}$-in. coarse aggregate is used, a saving of \$.47 per cubic yard. I think it desirable, therefore, that grading limits be inserted in the specifications to suit the construction requirements and economy of the situation. Moreover, these requirements had better be restricted within comparatively narrow limits to promote the greatest uniformity in the concrete mix; otherwise the proportions must be constantly adjusted to maintain a constant workability with a constant water ratio.

I am also glad to note that Mr. Chapman has emphasized the very great importance of recognizing the effect of moisture in bulking the sand. Engineers have been slow to allow for this phenomenon even though they knew it existed, probably through an unfounded fear that too much fine aggregate might result in their concrete. Again, I wish to voice my appreciation for Mr. Chapman's analysis of the concrete specification situation as it exists to-day.

Mr. Lesley. MR. ROBERT W. LESLEY.[1]—In this very excellent paper, Mr.

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[1] President, Surface Combustion Co., Philadelphia, Pa.

Mr. Lesley. Chapman suggests that there should be new kinds of cement and that "instead of trying to incorporate in a single cement all of the qualities demanded by all classes of service, might it not be cheaper and better to vary the cement to suit the service?" Now I should like to call attention to the fact that as far back as 1890 there were in existence a number of varieties of cement, such as portland cement, natural cement, improved cement, mixed portland cement and pozzuolana cement. At that time a cement specification had not been developed, although the American Society of Civil Engineers through a committee had presented a suggested method under which tests were supposed to be made. I recall presenting before the Engineers' Club of Philadelphia in 1898 a paper in which were tabulated the requirements of some 200 specifications which by that time had come into the laboratory of our cement works. Out of these 200 specifications there were hardly any two that agreed, and in each case they represented the desires and honest thought and wishes of the various engineers who wrote them. Thus each engineer had his own idea of the particular specifications for cement required to meet his needs. Instead of a definite standard to work to, the cement mill at that time had to consider the requirements of these many specifications and, of course, it was evident that no mill could possibly carry on intelligent, safe and practical business if it had to meet these various desires and indeed sometimes the whims of the engineers.

This in a word was the situation that faced our Society at its organization in 1902 and it is to be recalled that it was at the annual meeting in that year that the Committee on Cement was created, whose application to this matter of cement testing and specifications throughout the years that have passed has resulted in our present single standard specification for portland cement. It is interesting to follow the analogy of law making that a principle to be observed in the making of a law is to determine first the evil it is intended to correct. In 1902, what was the evil in the cement industry to be remedied by our Society for Testing Materials? The evil was these couple of hundred specifications. What was the remedy? The remedy was a consensus of the best thought of the best engineers in the country representing the various interested groups, and out of that consensus of thought came the remedy, namely, a single standard specification. Thus the evil was corrected and the new law came into life.

Now, when you come to think of what might occur if we had the production of new kinds of cement as suggested by the author, you can see what a hard road consumers and manufacturers would have

Mr. Lesley. to travel. In view of the past experience I am sure that Mr. Chapman does not propose to open that door now. He does not propose to give us "57 varieties" of cement for us all to wrestle with again. It is hard even to picture to you the ultimate result, but this is so well pictured in the next to last paragraph of Mr. Chapman's paper that by reading it you will readily see what might happen. Referring to the mixing of concrete, he says:

> "Occasionally an engineer wants a particular type of mixer and thinks none other as good. Another may want an abnormally long mixing time or may require that the other ingredients be mixed dry before water is added. In general the requirements on mixing appearing in the specifications of the Joint Committee seem satisfactory and present no particular problem. It will not be further discussed here."

If Mr. Chapman is correct in his statement of the case in the matter of mixing, you can see how much more difficult the case might be in the matter of the type of cement to be used. Recognizing the value of what Mr. Chapman has presented to us and the care with which his paper was prepared, I am sure that after considering what I have said he and I in the future will agree in this matter of cement without a question.

Mr. Thompson. MR. SANFORD E. THOMPSON.[1]—The paper presented, as has been indicated, is an outcome of conferences in Committee C-9 and its subcommittees, at which problems were brought forward and discussed by the various members. It contains most valuable material which will form an excellent basis for the consideration of various points that must be covered by the specification. One aim of the paper also has been to call attention to the disputed features in concrete specifications. It is our desire that we receive from members of this Society suggestions and comments that will aid the committee in framing a satisfactory specification.

Mr. Ferguson. MR. L. R. FERGUSON.[2]—This paper of Mr. Chapman, as Mr. Thompson has indicated, was prepared primarily with the idea of stimulating thought in connection with the preparation of a more adequate specification for concrete than we now have. It seems to me that the questions Mr. Chapman has raised are indications of a very significant trend in the technique of concrete preparation. One of the ingredients in concrete, namely, cement, has for a long time received an immense amount of attention and is receiving perhaps more to-day than ever before. If the same amount of attention is given to the other ingredients which go into the making of concrete,

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[1] President, The Thompson and Lichtner Co., Inc., Boston, Mass.

[2] Manager, Louisiana Portland Cement Co., New Orleans, La.

Mr. Ferguson.

to the way in which those ingredients are put together and the methods used in placing the concrete, we will naturally have a vastly improved product. As Mr. Goldbeck says, some of the problems involved in determining the proper qualities of aggregates are difficult of solution, but Mr. Chapman's paper shows that there is a tendency to give a greater amount of attention to the subject than ever before. The growing interest and increased acceptance on the part of engineers of the water-cement ratio is another very significant indication. The "factor of ignorance" is being removed to a greater and greater extent from concrete. Increased knowledge on the part of the engineer of the character of the materials he is employing and the method of putting them together, is making it possible to design and manufacture concrete so as to give the results which the engineer has anticipated in the design of his structures.

Mr. Kelley.

MR. F. W. KELLEY[1] (*by letter*).—Those who suggest the need for a large variety or number of grades of cement lose sight of one of the most important features of portland cement. Portland cement is only useful when it is mixed with aggregates and water to form concrete. This mixing may be done in ways to secure a variety of results with standard portland cements. What the user really wants is a variety of concrete; and this is best secured by varying the other components used with a standard portland cement. The methods to be used in securing these different results in concrete are now well known, or may readily be learned. The wide variety of concrete thus available would appear to meet every need.

Unusually high early-strength concrete, or concrete mortars of little strength, can be made with portland cement and in general at a lower cost than with special cements. Our present knowledge of the art seems to indicate that only in extreme cases where the need for high 24-hour strength justifies the additional cost, are special cements warranted.

The commercial advantages of a standard portland cement giving a wide variety of results in concrete, compared with a large variety of special cements giving the same variety of results in concrete, must be obvious. If further evidence is needed, the statistics of manufacturing in the United States, just made available through the recent census, supply it. These show that the value of product produced per worker is 40 per cent greater in the large manufacturing plants in the United States than in the next smaller plants and 130 per cent greater than in the smallest plants. The effect upon the market price at which the commodity can be sold is obvious, and a cor-

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[1] President, North American Cement Corporation, Albany, N. Y.

Mr. Kelley. responding advantage to the user follows. A standard product permits of larger scale production.

Learning how to meet our needs with our present available and reliable portland cement will get us better results than dreaming of new products to get the same results. Whether we like it or not, making concrete is a manufacturing operation, and the unseen ingredients of brains and care will always have an important effect upon the resulting product.

Mr. Chapman. MR. CLOYD M. CHAPMAN (*author's closure by letter*).—Both Mr. Lesley and Mr. Kelley have warned us against more than one kind of cement. Mr. Kelley suggests that the characteristics required of a concrete to withstand the particular type of exposure to which it will be subjected may be obtained by varying the other components. To the extent that this is possible, well and good. But the other components must usually be those available near the work. The cement may be shipped from a distance but the aggregate found near the job must be used, though it is often not of the best for the purpose. The selection available is usually not large.

These questions are frequently asked: Can a cement be made which will build a better concrete pavement than our present standard portland cement? Can a cement be made which will better withstand the action of sea water, or of alkali? If the answers are "Yes," would it not pay to manufacture such cements? The extensive and intensive investigations now under way into the chemical and physical structure of cement may help us to answer these questions more definitely than they can be answered with our present knowledge.

CONCRETE IN TENSION

By A. N. Johnson[1]

Synopsis

This paper describes a series of tests on 144 specimens of mortar and concrete in tension and corresponding compression tests of mortar and concrete cylinders, the areas of the tension and compression specimens being the same, approximately 16 sq. in.

The object was to compare the ratio of strength in tension to the strength in compression. This ratio was found to be a constant for all mortars and concretes tested after a period of about 90 days, the value of the ratio being 0.08. For mortars and concretes less than 90 days old, the ratio was practically the same for the mortar and concrete, but varied with age from 0.15 to 0.08.

Special apparatus and procedure for testing the tension specimens were required and are described and illustrated.

A few data are given on modulus of elasticity of concrete in tension.

The tests here described are the result of cooperative work between the University of Maryland, the State Roads Commission of Maryland and the U. S. Bureau of Public Roads.

The purpose was to secure more data as to the relative strength of mortar and concrete specimens in compression and tension, the tension specimens to have a cross-sectional area equal to that of the compression specimens. To do this required special apparatus which was designed and made in the shops of the University of Maryland.

Test Specimens

Each tension specimen consists essentially of a cylindrical section 9 in. long by 4½ in. in diameter, on each end of which inverted frustums of cones were made, so that the total length of the specimen was nearly 21 in. The cylindrical portion of the specimen was the size of the cylinders used in the compression tests, 9 in. long by 4½ in. in

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[1] Dean, College of Engineering, University of Maryland, College Park, Md.

diameter. The tension test specimens were cast in brass molds which were split their entire length and bolted together. Before casting a specimen, the molds were rubbed with an oily rag. The concrete was put in from one end and tamped with a rod. While the concrete was being placed, the molds were hit with a wooden mallet which resulted in a concrete free from small air pockets.

The compression specimens were cast in steel molds which were made by splitting seamless tubing along one element of the cylinder and holding the edges together by means of bolts attached to lugs. Compression specimens were tamped as the concrete was poured in one end and the molds were also struck with a wooden mallet.

From a given batch of mortar or concrete, three compression and three tension specimens were made. No particular care was taken, however, that the various batches should have the same water ratio. The sand was quartz containing 4 to 5 per cent of silt, wet measure, with a trace of inorganic impurities, and had a fineness modulus of 3.28. The coarse aggregate was limestone not exceeding 1 in. in size, having a fineness modulus of 6.20. The mortar specimens were made up of 1 part of cement to 2 parts of sand. The concrete specimens were made up of 1 part of cement, 2 parts of sand, and 3 parts of coarse aggregate.

The specimens were stored in damp sand for about two weeks and then removed to a room in the laboratory. They were tested at various ages up to eight or nine months.

METHOD OF TESTING

In the design of the apparatus to hold the tension specimen, special care was taken to insure that the geometric axis of the tension specimen should coincide with the line of force applied to the specimens by the testing machine. This was accomplished as follows: in preparing the tension specimens for test, the cylindrical portion was surrounded by a split aluminum cylinder, held in place by a wooden clamp. Brass ends were made so that the opening was sufficiently large to go over the head of the tension specimen. These brass ends were split and bolted together when ready for use. They rested upon the aluminum cylinder by means of which it was possible accurately to center them with reference to the axis of the specimen. There was thus left an annular space between the brass end piece and the specimen which was filled with melted rosin poured through holes at the top. As soon as both end pieces had been thus placed, the aluminum cylinder surrounding the center portion of the specimen was removed. Reference to Figs. 1 and 2 will show the arrangement of the specimen

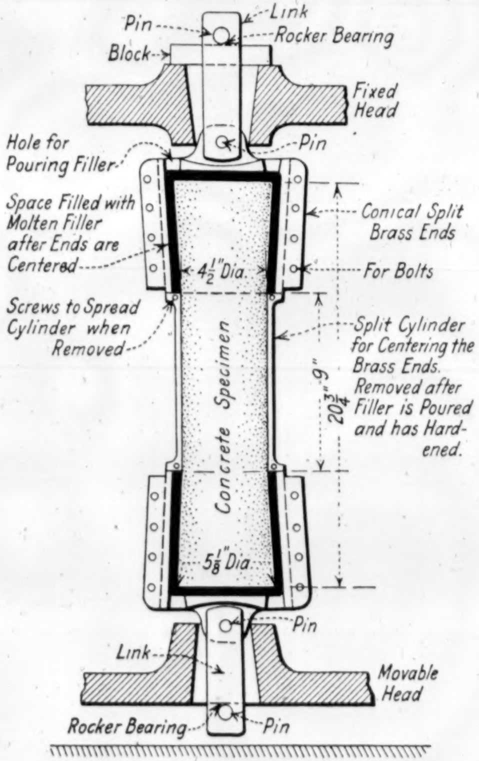
Link
Pin
Rocker Bearing
Block
Fixed Head
Pin
Hole for Pouring Filler
Space Filled with Molten Filler after Ends are Centered
Conical Split Brass Ends
$4\frac{1}{2}''$ Dia.
For Bolts
Screws to Spread Cylinder when Removed
Split Cylinder for Centering the Brass Ends. Removed after Filler is Poured and has Hardened.
9"
$20\frac{3}{4}''$
Concrete Specimen
$5\frac{1}{8}''$ Dia.
Pin
Link
Movable Head
Rocker Bearing
Pin

FIG. 1.—Arrangement of Concrete Specimen for Tension Tests.

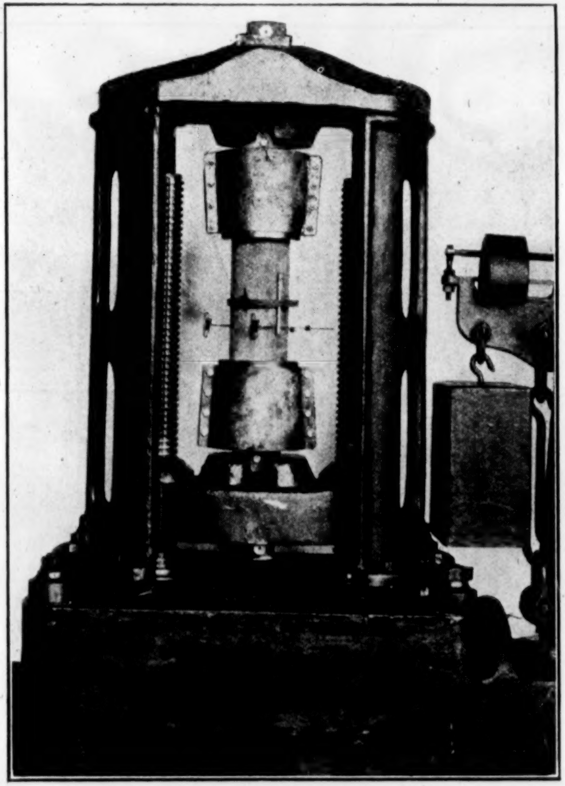

FIG. 2.—Tension Test Specimen of Concrete Ready for Test.

Note mirror extensometer also in place.

and the apparatus. The diagram shows clearly how, by means of links and pins with rocker bearings, the specimen was mounted in the machine in such a manner that there was no twist or eccentric loading given the specimen during the application of the load. A 100,000-lb. Riehlé machine was used for these tests, the load being applied slowly by hand. In operation, the apparatus worked very satisfactorily.

TABLE I.—RESULTS OF COMPRESSION AND TENSION TESTS OF CEMENT MORTAR (MIX 1:2).

Cross-sectional area 16 sq. in. (approximately). Each result, average of 3 specimens.

Age, days	Compressive Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Ratio, Tensile to Compressive Strength	Age, days	Compressive Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Ratio, Tensile to Compressive Strength
20	2510	349	0.14	162	6123	399	0.07
47	3827	421	0.11	167	6750	431	0.06
55	2640	442	0.17	173	7133	466	0.07
76	4990	434	0.09	184	5790	525	0.09
85	4200	370	0.09	191	6340	630	0.10
137	6230	347	0.06	198	5420	494	0.09
139	7140	489	0.07	199	5873	496	0.08
141	7183	434	0.06	212	5613	541	0.10
151	7577	488	0.06	253	6663	547	0.08
156	7223	583	0.08	255	6183	505	0.08

TABLE II.—RESULTS OF COMPRESSION AND TENSION TESTS OF CEMENT CONCRETE (MIX 1:2:3).

Cross-sectional area 16 sq. in. (approximately). Each result, average of 3 specimens.

Age, days	Compressive Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Ratio, Tensile to Compressive Strength	Age, days	Compressive Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Ratio, Tensile to Compressive Strength
18	1297	...	....	181	1847	155	0.08
33	2160	349	0.16	185	4280	326	0.08
50	3197	335	0.10	192	3773	301	0.08
69	3043	360	0.12	194	4667	307	0.07
83	3007	220	0.07	196	2130	192	0.09
90	2503	126	0.05	197	2863	235	0.08
140	3353	334	0.10	200	2503	236	0.09
146	4160	369	0.09	215	4260	330	0.08
154	4175	293	0.07	225	3337	298	0.09
159	3310	244	0.07	239	3647	284	0.08
165	3473	257	0.07	243	2797	247	0.09
172	3023	221	0.07	249	3373	248	0.07

Practically all the concrete tension specimens broke within or near the middle third of the cylindrical portion of the specimen, while a greater number of the mortar tension specimens broke near one clip or the other. In all instances, the breaks were nearly planes perpendicular to the axis of the specimen.

The compression tests were made with a spherical bearing block and usually a very thin cap of plaster was applied to the ends to insure flat bearings.

TEST RESULTS

The results obtained are detailed in Tables I and II. Table I shows the results for the mortar specimens, and Table II for the concrete specimens. It will be noted, first, that the ratio of tensile to compressive strength is practically the same for the mortar specimens as for the concrete specimens; and, second, that for specimens up to 90 days old the ratio in each case varies from 0.15 to 0.08, and after this period, the ratio becomes practically constant at 0.08. Thus, there is not the proportionate increase in tensile strength due to the age of the concrete as is the case with compression during the first 60 to 90 days, but beyond this time it appears that both are modified in practically the same ratio. These relations are brought out clearly in Fig. 3.

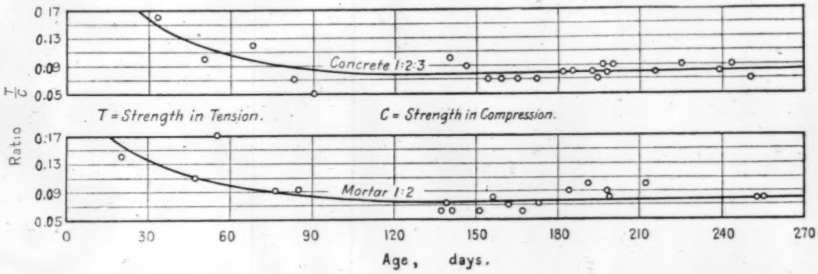

FIG. 3.—Relation Between Ratio of Tensile Strength to Compressive Strength and Age of Concrete.

The agreement of the results seems so persistent, even for considerably wide variation in the character of the concrete, that there exists considerable definite evidence as to the ratio between the tensile strength and compressive strength. Thus, in any given case the compressive strength being determined, the tensile strength is known with sufficient accuracy for most purposes.

The lack of any attempt to control the water ratio and the comparatively short period of curing accounts for the irregularity in the results when compared with the age of the specimens. As it was the intent primarily to study the ratio of tensile to compressive strengths, it was not necessary, in fact probably better, that there be a variety in the specimens that would correspond with variations that might be expected in most commercial work.

MODULUS OF ELASTICITY OF CONCRETE IN TENSION

During these experiments, a few measurements were made to determine the elastic curve of the concrete in tension. It is, perhaps, sufficient here to state a few of the results obtained. The method of observing the deformations in tension was by means of a mirror extensometer attached (see Fig. 2) in a manner similar for observations of compression cylinders which was fully described in a previous paper by the author.[1] As illustrative of some of the results obtained, attention is called to Fig. 4, which shows the deformations obtained upon a 1:2 mortar specimen, which broke at 340 lb. per sq. in., and indicates a modulus of elasticity of 4,200,000 lb. per sq. in. The values of *E* for the few specimens tested varied from 3,300,000 to over 5,000,000 lb. per sq. in. for some specimens.

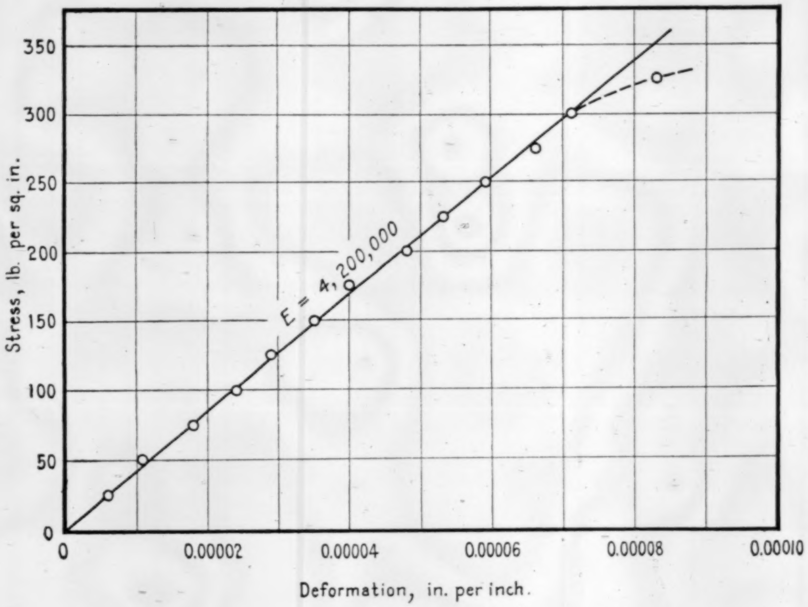

FIG. 4.—Stress-Strain Diagram of Cement Mortar (1:2) Cylinder in Tension.

Age, 67 days. Broke at 340 lb. per sq. in., at center.

It is the expectation to carry on a more extended series of observations of the modulus elasticity of concrete in tension, particularly with alumina cement.

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[1] A. N. Johnson, "Direct Measurement of Poisson's Ratio for Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 1024 (1924).

DISCUSSION

Mr. Proudley.

MR. C. E. PROUDLEY[1] (*presented in written form*).—In our studies of concrete in tension at the U. S. Bureau of Public Roads, a pair of grips built after the design of Mr. D. A. Abrams of Lewis Institute was used. These grips consist of two pieces of 6-in. steel pipe lined with leather and split part way along four elements so as to slip over the ends of a straight cylindrical concrete specimen. The segments are then drawn tight by means of tangential bolts so that enough friction is developed between the concrete and leather to prevent slipping during the application of the load. Ball-and-socket joints in each grip make the set-up self aligning.

The tension specimen is a cylinder 21 in. long by approximately 6 in. in diameter. These are molded in the same manner as compression specimens except that the successive layers of concrete were 5 or 6 in. deep instead of 3 or 4 in. The grips cover about 5 in. of each end of the specimen, leaving about 11 in. clear between grips for the break.

During these tests, it developed that the majority of specimens broke near enough to one or the other of the grips to permit a second test on the same specimen. The clear section for the second break was usually about 5 in. and in a very few instances only 4 in.

Naturally enough, the second break usually gave a higher result than the first, probably due to lack of homogeneity of the specimens. There seems to be an advantage in taking the average of the first and second breaking strengths as the true tensile strength of the entire specimen in that the results of tests of several specimens are apparently more consistent. For example, in one series of 7-day tests the percentage difference between strength of specimens was found to be 9.0 per cent when first breaks alone were considered and this was reduced to 4.3 per cent when the average of first and second breaks was taken as the strength of the specimen. For leaner concrete the advantage is more marked. At the age of 46 days, however, the effect of this procedure is much less than at 7 days, as is seen in Table I.

The test results given here are of concrete made of Potomac River gravel 1¼ to ¼ in., and Potomac River sand passing ¼ in. of medium grading.

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[1] Assistant Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

Mr. Proudley.

Note, however, the difference between the first and second break on the same specimen. In the case of the 1:2:4 concrete it averages 23 per cent and for the 1:3:6 concrete 31.5 per cent. No doubt these variations could be reduced by the use of slower rate of load application. The load was applied in these tests by means of a 100,000-lb. universal testing machine with the moving head traveling 0.1 in. per minute. At this rate a high personal factor was introduced, especially in the weaker specimens.

TABLE I.—TENSILE STRENGTH OF CONCRETE.

Age, 46 Days

Specimen	Water-Cement Ratio	First Break, lb. per sq. in.	Variation from Average		Second Break, lb. per sq. in.	Average	Variation from Average	
			Pounds	Per Cent			Pounds	Per Cent
1:1½:3 CONCRETE								
No. 757....	0.70	297	3	1.0	286	292	1	0.3
No. 758....	0.70	310	16	5.4	247	279	14	4.8
No. 759....	0.70	275	19	6.9	340	308	15	5.1
Average.	....	294	13	4.4	291	293	10	3.4
1:2:4 CONCRETE								
No. 754....	0.90	262	15	6.1	294	264	8	2.9
No. 755....	0.90	257	10	4.0	336	296	24	8.8
No. 756....	0.90	223	24	9.7	291	257	15	5.5
Average.	....	247	16	6.6	307	272	16	5.7
1:3:6 CONCRETE								
No. 760....	1.10	146	22	17.7	178	162	18	12.5
No. 762....	1.30	112	12	9.7	175	145	1	0.7
No. 763....	1.30	115	9	7.2	134	125	19	13.2
Average.	....	124	14	11.5	162	144	13	8.8

TABLE II.—TENSILE STRENGTH OF CONCRETE.

1:2:4 Concrete

Cement	7 days			28 days		
	Tensile Strength, lb. per sq. in.	Compressive Strength, lb. per sq. in.	Ratio, per cent	Tensile Strength, lb. per sq. in.	Compressive Strength, lb. per sq. in.	Ratio, per cent
A....................	126	1 092	11.5	198	2 100	9.4
B....................	112	1 300	8.6	212	2 428	8.7
C....................	130	1 402	9.3	224	2 005	11.2
D....................	132	1 430	9.2	231	2 680	11.6
E....................	146	1 530	9.5	244	2 700	9.0
F....................	160	1 830	8.7	222	2 775	8.0
G....................	144	1 349	10.7	227	2 160	10.5
H....................	202	1 940	10.4	246	2 462	10.0
Average..............	144	1 484	9.7	225	2 414	9.8

Mr. Proudley.

Mr. Johnson has shown in his paper that at early periods the ratio of tensile to compressive strength of stone concrete and mortar is about 0.15, decreasing with age to about 0.08 after 90 days. The tests made at the Bureau of Public Roads do not confirm this finding entirely. The results given in Table II show a fairly constant ratio of nearly 0.10 at both 7 and 28 days. This may be due to the difference in materials used as the coarse aggregate or the difference in the test procedure. Each result in Table II is the average of two specimens broken twice.

The modulus of rupture is usually considered to be indicative of the tensile strength of concrete. The data in Table III give this comparison for the tests at the Bureau of Public Roads, and indicate that the modulus of rupture in pounds per square inch is about twice the actual resistance of the concrete to failure in direct tension. It should be explained, however, that the modulus of rupture is obtained on a 6 by 6 by 30-in. beam broken by the cantilever method developed by Clemmer and Burggraf.[1] From the meager data available it is indicated that the values obtained with the cantilever device are lower than those obtained with third-point loading. If this is true, it would reduce the ratio of tensile strength to modulus of rupture to some extent.

TABLE III.—RESULTS OF TESTS, SHOWING MODULUS OF RUPTURE.

1:2:4 Concrete

Cement	7 Days			28 Days		
	Tensile Strength, lb. per sq. in.	Modulus of Rupture, lb. per sq. in.	Ratio, per cent	Tensile Strength, lb. per sq. in.	Modulus of Rupture, lb. per sq. in.	Ratio, per cent
A	126	211	59.7	198	330	60.0
B	112	230	48.7	212	384	55.2
C	130	249	52.2	224	338	66.3
D	132	247	53.4	231	392	58.9
E	146	276	52.9	244	395	61.8
F	160	265	60.4	222	401	55.4
G	144	261	55.2	227	391	58.1
H	202	372	54.3	246	432	57.0
Average	144	264	54.6	225	383	59.1

The Bureau of Public Roads is at present making a study of the effect of type of aggregate, grading of fine aggregate and water-cement ratio on the ratio of tensile strength of concrete to the compressive strength at ages of 3, 7, and 28 days and 6 months, from which additional information of much interest is expected.

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[1] H. F. Clemmer and Fred Burggraf, "Transverse Testing of Concrete," *Proceedings*, Am. Concrete Inst., Vol. XXII, p. 304 (1926).

Mr. Goldbeck.

MR. A. T. GOLDBECK.[1]—It might be of some interest to point out the practical significance of the tensile strength of concrete, especially as applied to concrete roads. Let the accompanying Fig. 1 be the side view of a concrete road, and let *A* be an expansion joint. As long as the concrete is kept wet, it remains in an expanded condition, and just as soon as curing is stopped, the concrete begins to dry out and shrink. As it shrinks, end *A* moves over the subgrade and forces of friction are developed between the slab and the subgrade.

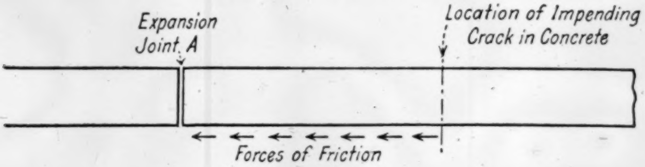

FIG. 1.—Diagrammatic Sketch of Side View of Concrete Road.

Because of the forces of friction, tension is produced in the concrete equal to the total friction existing between the expansion joint and any particular section we might consider. If a crack is impending at any section, the tensile strength of the concrete will equal the total friction existing from that section to the expansion joint, and the higher the tensile strength, the longer that length will be. It is thus very important that high tensile strength be attained in concrete for use in concrete roads, for the higher the tensile strength the further apart will be the transverse cracks.

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[1] Director, Bureau of Engineering, National Crushed Stone Association, Washington, D. C.

EFFECT OF CALCIUM CHLORIDE ON TRANSVERSE STRENGTH OF CONCRETE CURED AT VARIOUS TEMPERATURES

By Leroy C. Stewart[1]

Synopsis

This paper presents the data obtained from tests of about 1800 concrete beams 6 by 6 by 36 in. to determine the effect of calcium chloride upon transverse strength, expressed as modulus of rupture. The beams were tested as cantilevers, in such a way that three test breaks were obtained from practically each beam.

The following variables were involved:

1. Six representative brands of portland cement, used separately;
2. One brand of commercial calcium chloride as admixture, to the extent of 0, 2, 4, 6 and 10 per cent of the weight of cement;
3. Six conditions of curing, four series at temperatures respectively of 15, 22, 30 and 70° F., and two series of specimens poured out-of-doors in early winter when alternate freezing and thawing conditions prevailed and cured out-of-doors.

The concrete was a 1:5.14 mix by volume, having a water-cement ratio of 0.86 by volume. Each batch, sufficient for two specimens, was mixed four minutes in a half-sack rotating-drum mixer. No surface covering or curing other than calcium chloride admixture was provided. Aggregate and water for concrete subjected to freezing temperatures were warmed before using, except in the two outdoor series.

Duplicate beams of each mix, except for the two out-of-doors series, were made about two weeks apart. The beams were tested at 7, 14 and 28 days, and 3, 6 and 12 months.

The tests show that the transverse strength of concrete cured at low temperatures is decidedly increased when calcium chloride is used as an admixture in addition to ordinary cold-weather precautions. The data also indicate that strength of concrete poured and cured at normal temperatures or in a season of alternate freezing and thawing is improved by use of calcium chloride.

Introduction

The use of calcium chloride as an admixture for accelerating the set and gain in strength of portland-cement concrete has been investigated and discussed considerably during the past few years. In most cases, however, its effect on compressive strength has been studied, using cylinders and cubes made in the laboratory. It was felt that highway and structural engineers would be interested in data from large scale experiments to determine the effect of calcium chloride on transverse strength of concrete, particularly when low temperatures prevailed. Consequently, the work herein described was undertaken.

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[1] Chemical Engineer, The Dow Chemical Co., Midland, Mich.

MATERIALS AND PROCEDURE

Cement.—Investigators have intimated that calcium chloride affects various brands of cement differently. Therefore, six commercial brands of portland cement, representative of different processes of manufacture, were used separately. These were not bought on specification and it was assumed that they would be typical of the commercial product which might be encountered under average conditions. A sample of each of the cements was subjected to the standard soundness test[1] and found to be acceptable. The only other test made on the cements was a chemical analysis and the data thus obtained, together with notes as to the process of manufacture, are given in Table I.

TABLE I.—CHEMICAL ANALYSIS OF CEMENTS AND PROCESS OF MANUFACTURE.

	BRAND A	BRAND B	BRAND C	BRAND D	BRAND E	BRAND F
Loss on Ignition, per cent	4.63	5.53	5.33	4.79	4.06	4.10
SiO_2, per cent	20.41	21.65	23.70	21.80	20.85	22.37
Fe_2O_3, per cent	3.84	2.76	2.40	1.96	2.72	3.28
Al_2O_3, per cent	6.42	5.60	4.68	4.64	7.10	5.46
CaO, per cent	59.40	58.38	61.98	60.15	61.70	59.10
MgO, per cent	3.49	4.43	1.25	4.42	2.14	3.79
SO_3, per cent	1.54	1.84	1.73	2.50	2.07	2.81
Process	Semi-Wet	Dry	Wet	Dry	Dry	Dry
Raw Materials	Limestone and Clay	Limestone and Shale	Limestone and Clay	Limestone and Clay	Limestone and Slag	Marl and Clay

In commenting on chemical analysis, it is probable that the slightly high ignition loss values may be explained by the facts that brands A and E were obtained from dealers' warehouses and the others were received in less-than-carload shipments from the manufacturer's stocks, and that all the cement was stored for about two months before it was used. The chemical analyses were made at the time the cements were used.

Aggregate.—In order to insure uniformly high quality aggregate, a good grade of pit gravel was washed in a rotating drum with a heavy stream of water passing counter-current to the stone. The gravel from the washer was spread on steam coils to dry, after which it was screened into six fractions, the maximum size of piece used being 1½ in. in diameter. The fractions were recombined in closely-woven burlap bags by weighing the necessary amount of each one to give a total of 95 lb. The screen analysis of the aggregate as used in the test specimens was as follows:

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[1] A.S.T.M. Standard Specifications and Tests for Portland Cement (C 9 – 24), 1924 Book of A.S.T.M. Standards.

Retained on 1½-in. sieve	0	per cent
Retained on ¾-in. sieve	17	"
Retained on ⅜-in. sieve	51	"
Retained on 4-mesh sieve	68	"
Retained on 8-mesh sieve	75	"
Retained on 14-mesh sieve	79	"
Retained on 28-mesh sieve	83	"
Retained on 48-mesh sieve	98	"
Retained on 100-mesh sieve	100	"
Fineness Modulus	5.71	

The concrete for the low-temperature experiments was mixed in a half-sack rotating mixer located in a shed close to the constant-temperature room in which the specimens were made. Inasmuch as the majority of the work was carried on during winter months, most of the aggregate was heated in the bags on steam coils before being hauled to the mixer. In this way it was possible to pour a comparatively warm mix in a room maintained at the desired low temperature. This procedure corresponded to the heating of aggregate, as is recommended in cold-weather work. However, to avoid too rapid setting, the aggregate was not heated where 10 per cent of calcium chloride was used.

Proportioning and Mixing.—The mix was proportioned so as to use 29.1 lb. of cement to 190 lb. of aggregate, which was a ratio of 1:6.53 by weight or 1:5.14 by volume. Two gallons of water or water containing calcium chloride were used with the above amounts of cement and aggregate, giving a water-cement ratio by volume of 0.86. All batches of concrete were mixed four minutes.

Consistency.—The proportions of water and cement were kept constant at all times because of the important effect of this relationship on the physical properties of concrete. However, consistency was determined with a flow table,[1] and, although the data obtained with this apparatus varied somewhat, all mixes containing not more than 6 per cent of calcium chloride could be classified as being of practical working consistency, since the flow varied between the limits of 170 and 240 for individual tests, and averaged 212 for the six brands of cement without the use of calcium chloride and 222 with 2 per cent of the admixture. With 4 per cent of calcium chloride, the average flow was 225 and with 6 per cent of admixture the average was 217. Ten per cent of calcium chloride produced a decided stiffening of the mix in several cases so that it was impractical to perform the test with every batch. The average of all tests made with this amount was 174. The data on consistency of concretes with no calcium chloride and with 2 per cent of calcium chloride are given in Table IV.

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[1] Illustrated in a paper by G. M. Williams, "Some Determinations of the Stress Deformation Relations for Concretes Under Repeated and Continuous Loading," ***Proceedings***, Am. Soc. Testing, Mats., Vol. XX, Part II, p. 233 (1920).

TABLE II.—TEMPERATURE DATA RELATING TO TEST SPECIMENS.

Series	Specimens	Average Temperature of Mix when Poured	Temperature During Setting and Curing for 28 Days		
			Maximum	Minimum	Average
I	7 and 14-day	40° F.[a]	......	......	15° F.
	28-day to 1-year	76° F.	......	......	15° F.
II	7 and 14-day	50° F.	......	......	22° F.
	28-day to 1-year	47° F.	......	......	22° F.
III	7 and 14-day	68° F.	......	......	30° F.
	28-day to 1-year	62° F.	......	......	30° F.
IV	7 and 14-day	74° F.	......	......	70° F.
	28-day to 1-year	77° F.	......	......	70° F.
V[b]	28-day to 1-year	43° F.	53° F.	15° F.	33° F.
VI[b]	28-day to 1-year	35° F.	38° F.	14° F.	26° F.

[a] This comparatively low temperature was due to a temporary shutdown of lines supplying steam to the coils used for warming the aggregate.

[b] Series V and VI were cured out-of-doors commencing with pouring in December and figures given are only for first seven days after pouring.

Calcium Chloride.—One commercial brand of calcium chloride complying with A.S.T.M. requirements[1] was used to the extent of 2, 4, 6, and 10 per cent of the weight of the cement. Specimens containing no calcium chloride were also made. A heavy solution of the chloride was prepared and the proper amount of this incorporated in the gaging water for the various mixes.

Test Specimens.—The test beams were 6 by 6 by 36 in. Duplicate test beams were made in all cases about two weeks apart, except for the two series of specimens (V and VI) cured out-of-doors. About 1800 beams were made. The forms were of wood and were oiled each time before use. The wet concrete was puddled into place with a piece of wood 4 in. wide and tapered from 1 in. thickness to about ¼ in. at one end. The surface was then smoothed with two or three passes of a trowel and left exposed. At the end of a week or ten days, when the beams had thoroughly hardened, they were removed from the molds and cured as explained below.

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[1] Tentative Specifications for Calcium Chloride for Dust Prevention (D 98 – 22 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 790 (1922).

Curing.—The test beams are classified in six series according to method of curing, as given in Table II. Beams of Series I to IV, inclusive, were cured at temperatures of 15, 22, 30 and 70° F., respectively. Beams of Series V and VI were made and cured out-of-doors in early winter when alternating freezing and thawing conditions existed.

The beams of Series I, II and III were cured in a well-insulated room about 10 by 22 ft. in area. Seven feet above the floor was a tray which extended within 8 or 10 in. of the walls on three sides and within a few inches of the wall on the fourth. About a ton of cracked ice was maintained on this tray. A couple of hundred pounds of flake calcium chloride were sprinkled over the ice about every twelve hours during the period at which a temperature of 15° F. was being maintained. Mixtures of common salt and calcium chloride were sufficient to maintain the temperatures of 22 and 30° F. The mean temperature seldom varied more than 3 or 4° F. above or below the desired temperature and then only for a period of a few hours when warm concrete was being poured. After the 28-day tests were completed, the remaining beams of these three series were removed to an unheated building having an average temperature of 60 to 70° F. but ranging from 20 to 90° F. depending on the season of the year. These beams therefore thawed out slowly.

All beams of Series IV were cured at 70° F. in another building during early autumn. The temperature in this case varied not more than 8 or 10° F. either way for the period of a month.

The beams of Series V and VI were made out-of-doors in early winter, and were cured entirely out-of-doors. Aggregate for these beams was not heated but used at outdoor temperatures, since the temperatures were sufficiently high during the day to give a mix appreciably above the freezing point of water and it was desired to ascertain whether the use of calcium chloride under these conditions might make it possible to eliminate the heating of the aggregate.

Testing.—Beams cured at ages up to 28 days 15, 22 and 30° F., and out-of-doors in cold weather, were thawed over steam coils immediately prior to testing. All other beams were tested as removed from storage. In testing, the beams were clamped with troweled side up on a concrete pier and a lever arm 6 ft. long fitted over the free

TABLE III.—EFFECT OF CALCIUM CHLORIDE AS ADMIXTURE ON TRANSVERSE STRENGTH OF CONCRETE CURED AT VARIOUS TEMPERATURES.

Modulus of rupture obtained from 6 by 6 by 36-in. concrete beams subjected to cantilever load.
Mix 1:5.14 by volume (equivalent to 1:2:4). Water-cement ratio, 0.86 by volume.
Specimens tested at ages greater than 28 days removed from low temperatures at 28 days age.
Test specimens received no surface covering or curing other than calcium chloride as admixture.
Each value is average of three tests on each of two specimens made on different days.
Strength-ratios are percentages of strength of concrete of same history but without calcium chloride.
Calcium chloride expressed as percentage of weight of cement.

Brand of Cement	Series I Curing Temperature 15° F.			Series II Curing Temperature 22° F.			Series III Curing Temperature 30° F.			Series IV Curing Temperature 70° F.		
	Modulus of Rupture, lb. per sq. in.		Strength Ratio, per cent	Modulus of Rupture, lb. per sq. in.		Strength Ratio, per cent	Modulus of Rupture, lb. per sq. in.		Strength Ratio, per cent	Modulus of Rupture, lb. per sq. in.		Strength Ratio, per cent
	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride		0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride		0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride		0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride	
7-DAY TESTS												
A	45	102	227	197	228	116	225	263	117	270	351	130
B	44	89	202	158	137	86	199	157	79	311	307	99
C	35	65	185	112	108	96	171	149	93	322	274	85
D	19	42	220	133	135	101	165	157	95	302	342	113
E	19	61	331	178	194	109	213	213	100	322	315	98
F	19	79	416	146	126	86	200	152	76	316	344	109
Average	30	73	264	154	155	99	196	182	93	307	322	105
14-DAY TESTS												
A	38	126	331	240	259	108	308	294	95	329	379	117
B	48	75	156	198	182	95	259	216	83	351	342	97
C	18	64	355	149	127	85	210	184	88	339	332	98
D	19	39	205	132	193	146	223	192	86	346	427	123
E	15	64	426	187	223	119	301	258	86	362	389	107
F	19	93	489	192	168	87	234	202	86	377	389	103
Average	26	77	244	183	192	107	256	224	87	351	376	107
28-DAY TESTS												
A	126	167	133	197	229	116	398	362	91	391	524	134
B	85	179	211	100	227	227	318	291	92	367	447	122
C	62	142	229	45	169	376	293	291	99	382	380	99
D	32	180	563	73	211	389	255	250	98	341	427	125
E	24	107	445	106	235	221	341	341	100	337	390	116
F	18	122	678	80	168	210	312	282	90	459	492	107
Average	58	150	377	100	207	240	319	303	95	379	443	117
3-MONTH TESTS												
A	356	460	129	454	438	97	587	629	108	441	513	116
B	269	505	188	235	402	171	500	478	96	425	417	98
C	287	357	124	275	392	143	484	517	107	409	447	109
D	340	544	160	276	460	167	447	443	99	415	483	116
E	303	320	106	275	488	177	588	535	91	425	397	94
F	270	420	155	351	387	110	495	490	99	460	514	112
Average	304	434	144	311	428	144	517	515	100	429	462	108
6-MONTH TESTS												
A	502	595	119	480	682	142	594	675	114	426	519	122
B	375	513	137	335	593	177	627	672	107	447	502	112
C	397	620	156	349	535	153	520	549	106	445	474	106
D	424	660	155	287	588	205	527	575	109	479	471	93
E	333	515	155	232	595	256	644	542	84	352	515	146
F	342	479	140	440	557	126	467	564	119	573	484	84
Average	396	564	144	354	592	177	563	596	107	457	494	111
1-YEAR TESTS												
A	...	567	...	369	654	177	582	602	103	529	517	98
B	417	562	135	414	534	129	523	558	107	526	555	105
C	307	614	200	212	457	215	481	587	122	505	484	96
D	337	549	163	...	552	...	507	514	101	504	511	101
E	440	547	124	377	517	137	585	544	93	405	449	111
F	437	567	130	432	502	116	549	595	108	504	517	102
Average	388	568	125	361	536	155	538	567	106	496	506	102

end, which extended out 9 in. from the edge of the pier. A bucket was hung from the end of the arm, and an even flow of water, controlled by a quick-acting lever valve, directed into it until the beam broke. The bucket and water were then weighed to determine the weight necessary to break the specimen. It was possible to obtain three tests from practically all beams. A few broke in handling, and on some occasions the test break extended back from the edge of the pier so as to permit only one additional test. The modulus of rupture was calculated by use of the proper formula, correction being made for the weight of the cantilever arm and the piece of over-hanging beam.

TABLE IV.—FLOW TEST DATA.

Calcium chloride expressed as per cent of weight of cement.
Values for each brand of cement in each series are averages of six tests, each made on different days.

Brand of Cement	Flow, Series I, Curing Temperature 15° F.		Flow, Series II, Curing Temperature 22° F.		Flow, Series III, Curing Temperature 30° F.		Flow, Series IV Curing Temperature 70° F.		Flow, Average Four Series	
	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride	0 Per Cent Calcium Chloride	2 Per Cent Calcium Chloride
A	219	213	208	237	217	232	227	223	218	226
B	218	223	233	240	206	223	222	227	220	228
C	200	209	211	227	202	208	205	214	205	215
D	208	219	205	211	206	208	207	223	207	215
E	214	232	206	228	208	227	222	240	213	232
F	213	220	211	226	203	202	201	210	207	215
Average	212	219	212	228	207	217	214	223	212	222

NOTE.—Consistency of concrete was measured by use of a flow table which consisted of a cone, 8 by 12 by 6 in., and a table, 30 in. in diameter, arranged with its metal top mounted on a cam in such a way that it could be raised and dropped $\frac{1}{2}$ in. The concrete was molded in the cone on the table and, immediately after filling, the conical mold was withdrawn and the table dropped 15 times in about 10 seconds. The base diameter of the concrete thus jigged was measured and expressed as a percentage of the original base diameter. This value is the "Flow" of the concrete.

DATA AND DISCUSSION

Effect of Calcium Chloride on Transverse Strength.—The strength data obtained for the six different brands of cement with no calcium chloride and 2 per cent of calcium chloride in concrete cured at the different temperatures are given in Table III. Consistency data for the mixes listed in Table III will be found in Table IV. The average effect of the different amounts of calcium chloride, used separately with six brands of cement at various ages and temperatures of curing, including out-of-door tests, is shown graphically in Figs. 1 and 2.

It will be noted that the maximum strength obtained was for year-old concrete containing 6 per cent of calcium chloride and cured for the first 28 days at 15° F. At the time this concrete was poured, the temperature of the mix was about 75 to 80° F., which enabled the accelerated set to take place before freezing could occur. It is

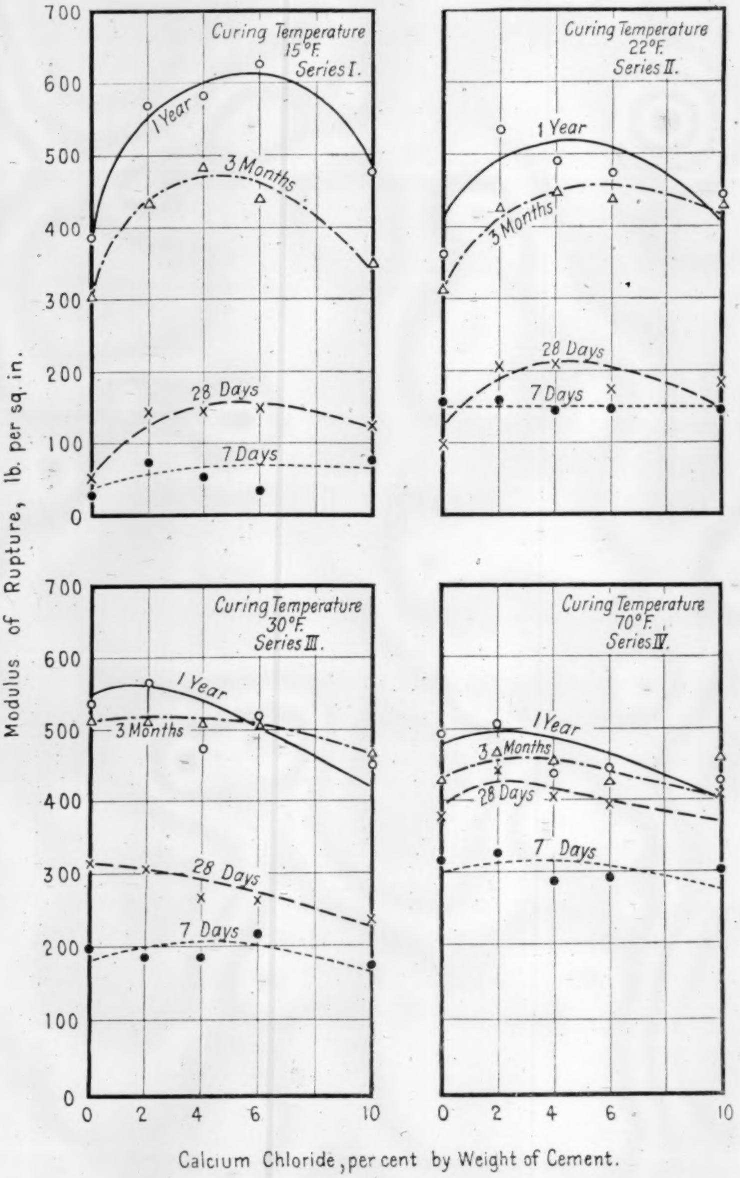

FIG. 1.—Effect of Calcium Chloride on Transverse Strength of Concrete as Influenced by Curing Temperature.

Specimens tested at ages greater than 28 days; removed from low temperatures at that age.
Test specimens received no surface covering, or curing other than calcium chloride admixture.
Each plotted value is average of 36 tests involving six brands of cement, duplicate specimens and three tests on each specimen.

also apparent that at temperatures above 15° F., 2 to 4 per cent of calcium chloride gave maximum strengths.

Figure 1 indicates a decided increase of strength due to the use of calcium chloride in the concrete cured for 28 days at 15 and 22° F. Also, an appreciable benefit from the admixture is shown in the curves for concrete cured at 70° F. The mixes cured at 30° F. show practically the same strengths without calcium chloride as with 2 and 4 per cent of the admixture. Except for the usual vagaries of physical testing, there is no apparent explanation for this different behavior at 30° F. than at lower or higher temperatures.

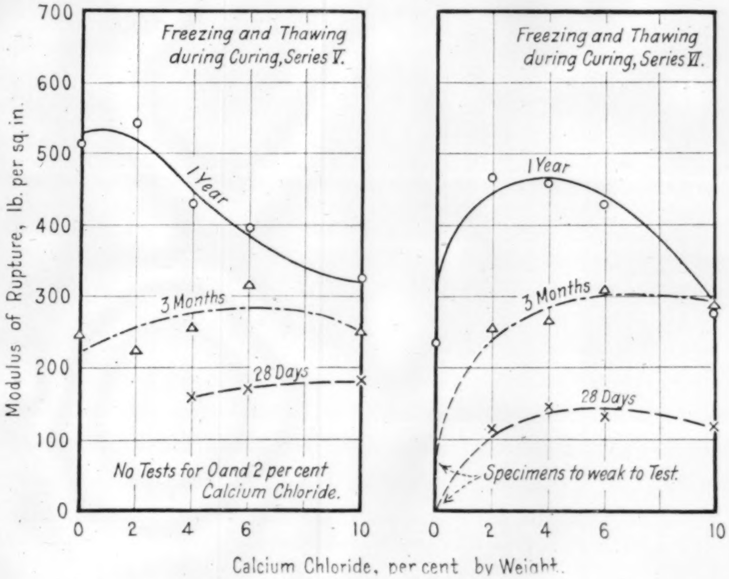

FIG. 2.—Effect of Calcium Chloride on Transverse Strength of Concrete Poured Out-of-Doors in Early Winter.

Specimens poured and aged out-of-doors. No covering, or curing other than calcium chloride as admixture.

Each plotted value is average of 18 tests involving six brands of cement and three tests on one specimen.

Temperatures during first seven days curing:

Series V Maximum 53° F., Minimum 15° F., Average 33° F.
Series VI Maximum 38° F., Minimum 14° F., Average 26° F.

In Fig. 2, showing the results for beams which were poured out-of-doors without warming of the aggregate, and exposed from the beginning to varying conditions of freezing and thawing, as well as rain, sleet, and snow, it is not surprising that similar mixes of the two series do not show equal strengths or that specimens in the same series with varying amounts of calcium chloride do not exhibit a

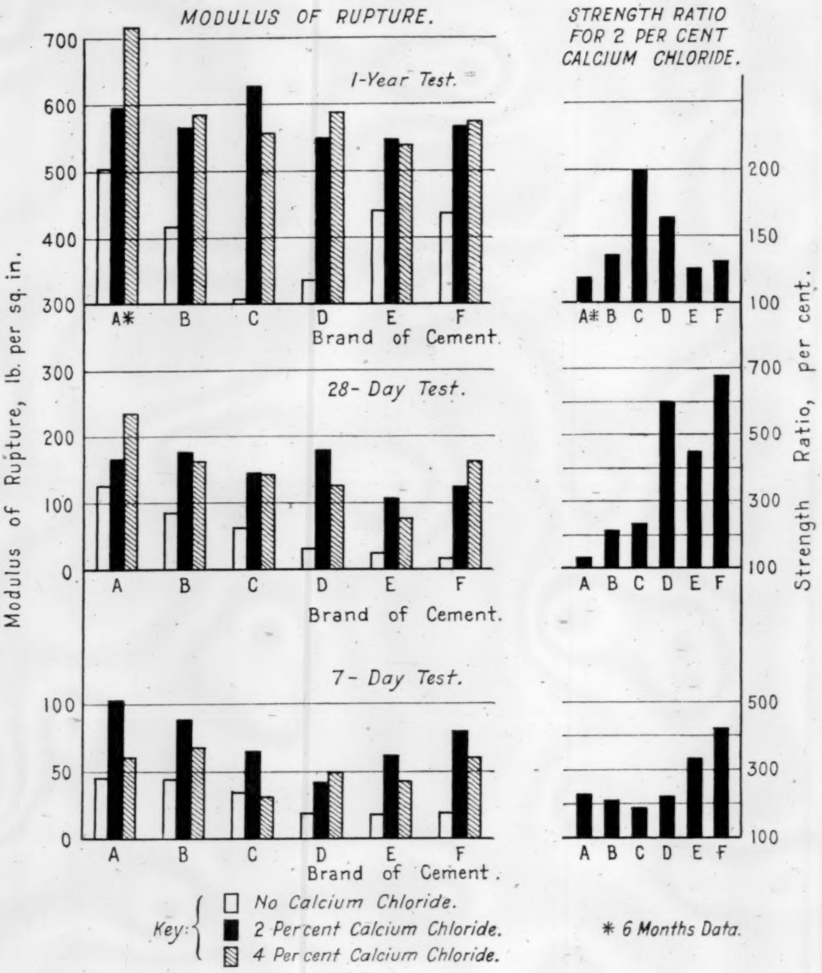

FIG. 3.—Effect of Calcium Chloride on Transverse Strength of Concrete Cured at 15° F., as Influenced by Brand of Cement.

Specimens tested at ages greater than 28 days, removed from low temperatures at that age.

Test specimens received no surface covering or curing other than calcium chloride admixture.

Each plotted value is average of 6 tests involving three tests on each of two specimens made on different days.

Strength-ratios are for mixes containing 2 per cent of calcium chloride and are percentages of strength of concrete of same history but without calcium chloride.

Calcium chloride is expressed as per cent of weight of cement.

regularity in effect of the admixture. It would be expected, for example, that specimens made in the morning and exposed to thawing temperatures during the day would not be subjected to as detrimental conditions during setting as beams made in the late afternoon at a time when freezing temperatures were beginning to prevail. Perhaps the most significant fact in the outdoor cold weather experiments is that many test specimens which did not contain any calcium

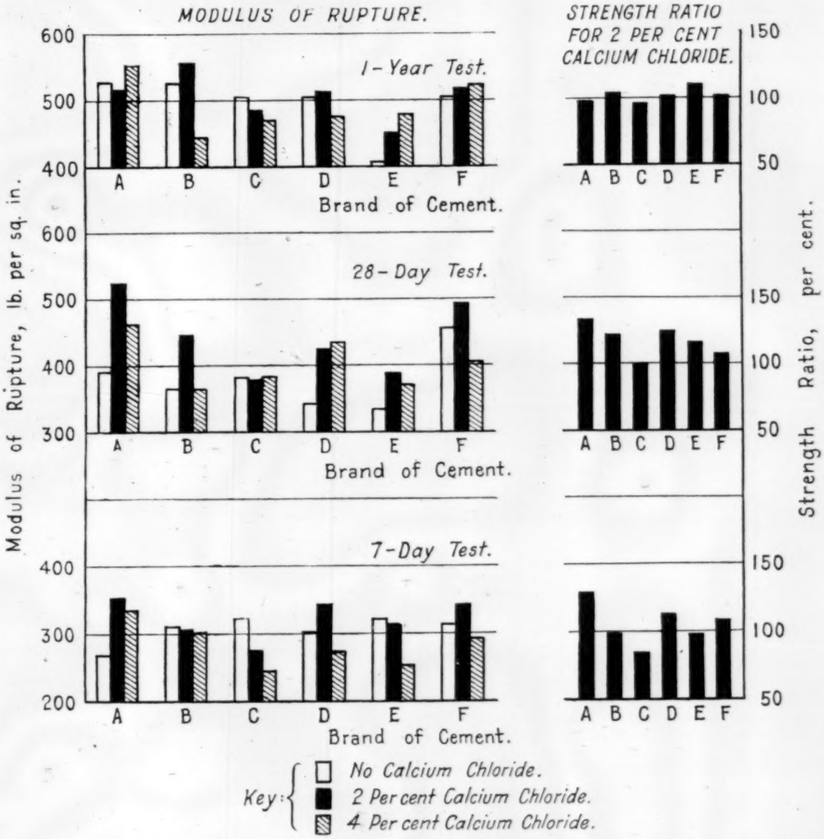

FIG. 4.—Effect of Calcium Chloride on Transverse Strength of Concrete Cured at 70° F., as Influenced by Brand of Cement.

Specimens tested at ages greater than 28 days, removed from low temperatures at that age.
Test specimens received no surface covering or curing other than calcium chloride admixture.
Each plotted value is average of 6 tests involving three tests on each of two specimens made on different days.
Strength-ratios are for mixes containing 2 per cent of calcium chloride and are percentages of strength of concrete of same history but without calcium chloride.
Calcium chloride is expressed as per cent of weight of cement.

chloride disintegrated during weathering or in the handling incident to testing. As a result of these experiments, it is evident that, with or without the use of calcium chloride, ordinary cold-weather precautions should not be omitted when concrete is poured in a season of alternate freezing and thawing conditions, even though the concrete might be subjected to non-freezing temperatures for the first few hours.

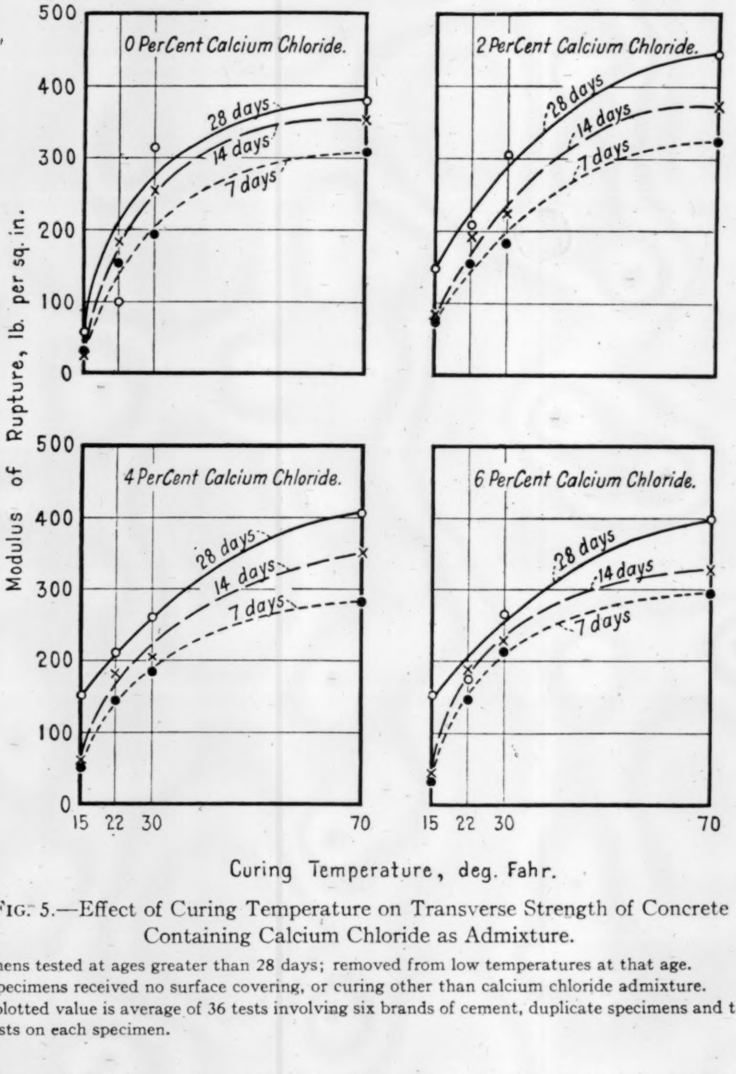

FIG. 5.—Effect of Curing Temperature on Transverse Strength of Concrete Containing Calcium Chloride as Admixture.

Specimens tested at ages greater than 28 days; removed from low temperatures at that age.
Test specimens received no surface covering, or curing other than calcium chloride admixture.
Each plotted value is average of 36 tests involving six brands of cement, duplicate specimens and three tests on each specimen.

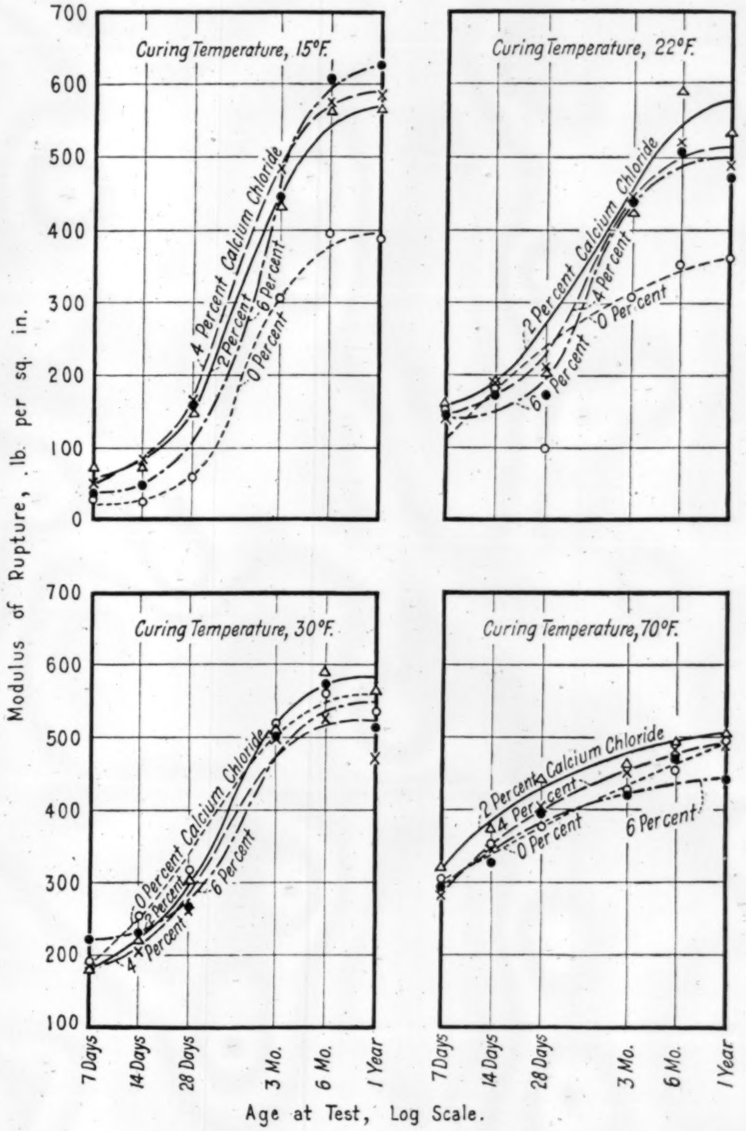

FIG. 6.—Effect of Age on Transverse Strength of Concrete Containing Calcium Chloride as Admixture.

Specimens tested at ages greater than 28 days; removed from low temperatures at that age.

Test specimens received no surface covering, or curing other than calcium chloride admixture.

Each plotted value is average of 36 tests involving six brands of cement, duplicate specimens and three tests on each specimen.

In Fig. 3 there are shown graphically the transverse strength data for concrete containing 0, 2 and 4 per cent of calcium chloride for the six different brands of cement at ages of 7 and 28 days and 1 year, in mixes cured for 28 days at 15° F. Figure 3 also shows the corresponding strength-ratios for mixes containing 2 per cent of calcium chloride. Figure 4 shows data similar to those of Fig. 3 for the curing temperature of 70° F. A study of these two figures, which represent the two temperature extremes investigated, as well as the data of Table III, shows that although calcium chloride seemed to affect the various brands of cement differently, its behavior with any one brand was not consistently the same in relation to its effect on the other brands, under all conditions of temperature and age.

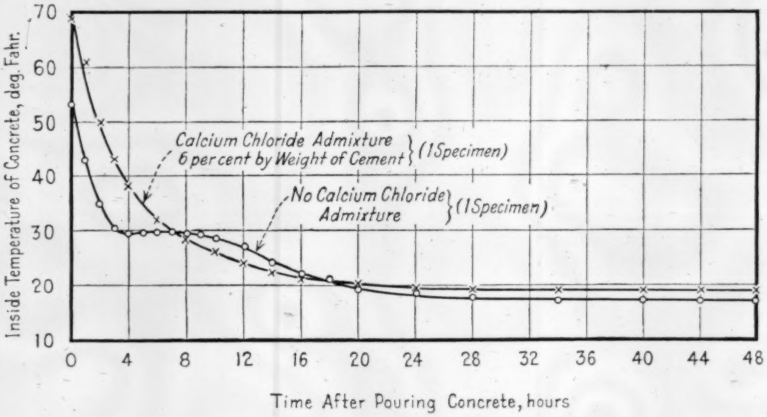

FIG. 7.—Temperatures Inside 6 by 6 by 36-in. Concrete Beams Cured at 15° F.

In view of this fact and also considering that an investigation is now in progress in which the behavior of calcium chloride with about sixty different cements is being studied, it is not considered advisable on the basis of the data given to attempt to draw any definite conclusion as to the effect of calcium chloride on strength of concrete as influenced by brand or chemical properties of the cement.

The graphical data of Figs. 3 and 4 would indicate, however, that different brands of cement are affected to a different extent by calcium-chloride admixture, and that this effect on transverse strength is quite pronounced at ages up to a year, for concrete poured and cured for at least a month at low temperatures. The data also indicate that for concrete cured at normal temperatures such as 70° F., the influence of brand of cement on transverse strength is appreciable at early ages, but almost negligible at one year.

Effect of Curing Temperature and Age.—The data obtained also yield information as to the effect of temperature on the development of transverse strength in concrete as well as the effect of age on increase in strength. Figure 5 shows graphically the increase in strength due to curing temperature, while Fig. 6 shows the gain in strength with age.

Effect of Calcium Chloride on Temperature Inside of Concrete Cured at 15° F.—By embedding the bulb of a recording thermometer in two test beams, poured and cured at 15° F., one with, and one without calcium chloride admixture, the interesting data shown graphically in Fig. 7 were obtained. A logical interpretation of the curves is that the concrete containing the calcium chloride acquired its natural set before freezing could take place as shown by the steady drop in temperature inside the specimen. In the beam containing no calcium chloride, the temperature during cooling paused at the point where freezing took place until all the free water had been converted to ice, before it continued down to the temperature of the room.

Although this experiment is not very comprehensive in scope, and will form the basis of a more extensive research program, it is offered with the idea that the data will be of such interest to other investigators that they will undertake similar experiments.

CONCLUSIONS

The principal conclusions to be drawn from this investigation are:

1. The transverse strength of concrete cured at low temperatures is decidedly increased when calcium chloride is used as an admixture to the extent of 2 to 6 per cent of the weight of cement, in addition to other common cold-weather precautions.

2. The transverse strength of concrete cured at normal temperatures is increased by use of 2 per cent of calcium chloride as admixture, based on weight of cement.

3. Concrete poured in early winter when freezing and thawing conditions prevailed, and cured entirely out-of-doors, was improved by addition of from 4 to 6 per cent of calcium chloride. In many cases, test specimens containing none of the chloride were too weak to withstand handling incidental to testing.

4. Although calcium chloride seemed to affect the various brands of cement differently, its behavior with any one brand was not consistently the same in relation to its effect on the other brands under all conditions of temperature and age.

DISCUSSION

Mr. Mattimore. MR. H. S. MATTIMORE.[1]—As I understood Mr. Stewart, the concrete cured in low temperatures showed a greater increase in strength with calcium chloride than it did at 70° F. This checks some work that we have carried on in our laboratory, but there is one feature I should like to bring out relative to curing. In our work on calcium chloride we found that the percentage of humidity was an important consideration. I should like to check Mr. Stewart's comparative curing of the plain specimens and of the calcium-chloride specimens.

Mr. Stewart. MR. L. C. STEWART.—Of course, at the low temperatures such as 15° F., the humidity of the air was quite low, but for the 70° F. series, which would be the one in which you would be particularly interested, the experiments were carried on inside a building where the humidity record showed a variation from 45 to 70 per cent, depending on the weather out-of-doors. We did not maintain a constant humidity during the whole course of the experiments. All the specimens were exposed to the air without any treatment other than the calcium-chloride admixture.

Mr. Mattimore. MR. MATTIMORE.—Do you refer to the calcium-chloride specimens or the plain specimens?

Mr. Stewart. MR. STEWART.—I refer to both kinds.

Mr. Mattimore. MR. MATTIMORE.—How about concrete which has been water cured? Did you not have any such specimens?

Mr. Stewart. MR. STEWART.—No, all specimens were air cured. Based on data which have been published, and from other data we are now obtaining in our investigations, the same increase in strength due to calcium chloride would be found in water-cured concrete as in air-cured concrete.

Mr. Mattimore. MR. MATTIMORE.—I would not consider that you are by any means obtaining 100 per cent efficiency in your curing of the specimens with no calcium chloride. Under such curing calcium chloride has a very decided advantage. In order to get the comparison the plain concrete should have efficient water curing, and could be used as a basis in comparing the concrete containing calcium chloride; but under the method used in your test the plain concrete did not have efficient curing, and therefore is valueless as a comparison.

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[1] Engineer of Materials, Pennsylvania State Highway Department, Harrisburg, Pa.

Mr. Stewart.

MR. STEWART.—We did not make a comparison at normal temperatures between wet-cured concrete without calcium chloride and dry-cured concrete containing the admixture. Such experiments would be of most interest to Mr. Mattimore since they would be applicable to road building. Such tests to be of most practical value should be made out-of-doors, where in average weather, rainfall and high humidity at night would provide a certain amount of damp curing for the specimens not covered with wet earth. The normal temperature experiments described in this paper are more comparable to conditions found in the construction of buildings where wet curing is frequently not provided.

Mr. Clemmer.

MR. H. F. CLEMMER.[1]—I believe that the curing of concrete with calcium chloride under conditions of high humidity as in a moist room is not as satisfactory as under outdoor conditions where there would be a change in humidity causing evaporation. The calcium-chloride specimens reported by the author did not have the advantage of curing conditions one would expect to have under outside conditions and actual construction.

TABLE I.

Reference	Method of Curing Beams	Water-Cement Ratio	Modulus of Rupture, lb. per sq. in.					Strength Ratio, Per Cent of Strength of Concrete Without Admixture				
			3 days	7 days	14 days	28 days	90 days	3 days	7 days	14 days	28 days	90 days
1	Air curing, no surface treatment.	0.84	295	345	365	385	435	100	100	100	100	100
2[a]		0.83	310	340	365	400	470	105	99	100	104	108
13	2 in. earth, wet 3 days	0.84	340[b]	435	490	505	565	100	100	100	100	100
14[a]		0.83	330[b]	385	435	485	540	97	89	89	96	96
11	2 in. earth, wet 7 days	0.84	...	445[b]	480	530	605	...	100	100	100	100
12[a]		0.83	...	340[b]	445	480	525	...	76	93	91	87
7	2 in. earth, wet 14 days	0.84	...	...	460[b]	540	595	...	...	100	100	100
8[a]		0.83	...	...	370[b]	445	545	...	...	80	82	92

[a] Two per cent calcium chloride by weight of cement used as an admixture in the concrete.
[b] Beams in this case were covered with wet earth 2 days less time than stated under method of curing.

I think the main thing this investigation brings out is that you can depend on calcium chloride as an accelerator for outside conditions under low temperatures, but that the effect on each brand of cement must be determined as well as the strength of test specimens made up with the accelerator.

Mr. Gonnerman.

MR. H. F. GONNERMAN.[2]—During the summer of 1924 our laboratory carried out an investigation on the curing of concrete at Sacramento, Calif., in cooperation with the California Highway Com-

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[1] Technical Adviser, Solvay Process Co., New York City.

[2] Associate Engineer, Research Laboratory, Portland Cement Association, Chicago, Ill.

Mr. Gonnerman. mission,[1] which included a few tests on the transverse strength of concrete containing an admixture of 2 per cent of calcium chloride by weight of cement. The test specimens were 7 by 10 by 38-in. concrete beams which were loaded at the third points of a 36-in. span at ages of 3 to 90 days. The beams were covered with wet burlap while in the forms and upon removal after 16 to 20 hours they were cured as indicated in the accompanying Table I.

It will be noted that approximately the same modulus of rupture was obtained from the air-cured concrete containing 2 per cent calcium chloride as from air-cured concrete without the admixture and that beams cured with earth wet for 3, 7 or 14 days showed lower strength for the concrete with the admixture than for that without. Although only one percentage of calcium chloride and one brand of cement was used in these tests, they indicate that for the materials used, the calcium-chloride admixture was less effective in the moist-cured than in the air-cured specimens.

Mr. Fulweiler. THE CHAIRMAN (*President W. H. Fulweiler*).[2]—I do not know very much about curing concrete, but I should like to know how you would cure concrete with water at 15° F.?

Mr. Abrams. MR. D. A. ABRAMS.[3]—Strange as it may seem, portland-cement concrete does cure at temperatures below the freezing point. Tests carried out at the University of Illinois by McDaniel[4] gave valuable information on this subject. Compression tests were made on 8 by 16-in. cylinders of 1:2:4 concrete using sand and crushed limestone aggregates. The specimens were cured at different temperatures and tested at 3 to 28 days. These tests showed about the following percentages of the 28-day strength of similar concrete cured damp at 70° F.

Curing Temperature, deg. Fahr.	Per Cent of 28-day Strength of Similar Concrete Cured at 70° F.			
	3 days	7 days	14 days	28 days
20	10	18	30	42
30	16	25	42	57
40	20	33	54	72

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[1] Complete details of tests are reported in *Bulletin 15*, "Studies of Curing Concrete in a Semi-Arid Climate," Structural Materials Research Laboratory, Chicago, Ill.

[2] Chemical Engineer, United Gas Improvement Co., Philadelphia, Pa.

[3] Portland Cement Association, Chicago, Ill.

[4] Engineering Experiment Station *Bulletin 81*, 1915; also *Proceedings*, Am. Concrete Inst., 1916.

Tests were not made at exactly the temperatures given, but these percentages represent the trend of McDaniel's tests. Specimens were about 6 hours old when exposed to low temperatures; specimens cured below freezing were allowed to thaw out before testing. **Mr. Abrams.**

It is interesting to note that at 30° F. the 28-day strength of this concrete was 57 per cent of normal.

THE AUTOGENOUS HEALING OF CONCRETES AND MORTARS

By Herbert J. Gilkey[1]

Synopsis

To place the phenomenon of autogenous, or self, healing of portland-cement concretes and mortars in the category of fully known and usable engineering knowledge would require an investigation much more comprehensive than that here recorded. The chemical action involved has been described by Abrams as a probable deposition of soluble compounds in and across cracks caused by overloading. Some have recorded instances of cracks actually disappearing and not re-appearing under subsequent increased loading.

The present tests do not touch the chemical aspects of autogenous healing. They cover mortars and concretes tested for ultimate compressive strength at various ages and re-tested from one to five times in a period up to eight months in some cases. Consideration is given to the following general range of variables and conditions:

Water-cement ratio varied;
Diameters of sand particles varied;
Variable curing conditions prior to first test at 28 days (part time in air);
Some variation of proportions;
Some variation in the kinds and gradings of aggregates.

Mortar specimens were largely of 2 by 4 in. in size and concretes were all 6 by 12 in. All but one series were very accurately controlled laboratory mixtures. That series was on field samples taken from rather poorly controlled job concrete.

Enough tests were made on specimens dry between test and retest to clearly indicate that except for a slight strength gain due to drying out, specimens do not heal autogenously unless kept moist. No data on these preliminary tests appear in the paper.

The general conclusion from all the tests made is that autogenous healing action follows the same general law as uninterrupted strength gain from continuous moist curing. Specimens salvaged without much visible damage will often develop later strengths approximately 100 per cent of what they would have had without previous tests. Healing takes place to some extent in even badly shattered specimens, but the strength may never attain that at the former test. Strong specimens (rich in cement, low in water, old or well cured) shatter so badly at ultimate load that few and often none can be salvaged for retest. The percentage recovery for such specimens is much lower than that for weak ones but the cause probably lies in the greater damage at former test. Mixtures as lean and as wet as those commonly used will often recover fully, under moist curing, following test or over-stress from other cause. In these tests

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[1] Associate Professor of Civil Engineering, University of Colorado, Boulder, Colo.

there was no apparent disappearance of the cracks from former test and failure usually continued along the same general lines as it started at the former test. The fact that only a few months, at most, elapsed between test and retest might account for the non-disappearance of cracks. Moreover no refinements beyond careful inspection and general remarks, upon type and extent of failure, were employed to detect possible disappearance of cracks. To adequately detect the disappearance of visible cracks, they should be carefully marked immediately after test. This was not done.

INTRODUCTION

The subject of autogenous healing of concrete first came to the author's attention several years ago during his connection with the Engineering Experiment Station at the University of Illinois. Specimens in the damp room there have shown constantly increasing strengths, when removed and tested to the ultimate strength, from time to time, over a period of years. The phenomenon had been noted with interest but in the press of other work no effort had been made to record and catalog the data of the tests.

D. A. Abrams[1, 2] has made several references to observations of this healing process under more or less favorable conditions. In one case attention is called to the disappearance of cracks in a highway bridge two months old that did not reopen three years later under a load six times as great as that which caused the cracks originally. In another case, eight-year cylinders, not shattered at first test, developed an average strength of 228 per cent of that at 28 days.

S. C. Hollister[3] tells of healing a wide crack in a newly poured arch bridge so completely that the location of the crack was entirely lost.

Data on short-time autogenous healing tests and a discussion of the phenomenon are given in a paper by the author, on "The Curing Conditions of Concretes and Mortars."[4]

In the light of the foregoing allusions to autogenous healing, and others not mentioned, the author can advance no claim to originality or discovery. This paper does contain, however, certain new material on the subject and it is hoped that the findings may be of sufficient value to justify placing them on record at this time.

The tests herein reported were not conducted solely or even primarily to obtain data upon autogenous healing. In this respect, the subject is an easy one to investigate, since retests may be made in

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[1] *Bulletin No. 71*, University of Illinois Engineering Experimental Station.

[2] "Test of a 40 ft. Reinforced Concrete Highway Bridge," ***Proceedings***, Am. Soc. Testing Mats., Vol. XIII, p. 884 (1913).

[3] "Question Box," ***Proceedings***, Am. Concrete Inst., (1926).

[4] Herbert J. Gilkey, "The Effect of Varied Curing Conditions on the Compressive Strength of Concretes and Mortars," ***Proceedings***, Am. Concrete Inst., 1926, Table 9.

connection with almost any investigation and autogenous healing data obtained as a by-product. In another respect, however, the subject is difficult to cover satisfactorily. It is important that the load be lifted just as soon as possible after the ultimate strength has been reached. Even then it is impossible to classify accurately the extent of structural damage. Sometimes (usually with weak mixtures) specimens may be tested to their ultimate strength without any visible injury.

Strong mixtures are very difficult to salvage. They either shatter explosively at the ultimate load, or do so immediately after the ultimate load has been applied. Often the machine is actually in reverse before the specimen shatters. A machine designed for quick release of load would provide opportunity to carry the investigation further than has yet been done for strong mixtures.

Specimens were carefully capped with neat cement a few hours after making and tested between metal plates. A spherical bearing block was used on top and specimens were very carefully centered. Records of kind and extent of damage, as determined by careful visual inspection, were kept. It must be recognized that the classification of failures involves a considerable element of personal judgment. When only a very slight indication of damage could be found, it was often impossible to say whether the weakening was caused by crushing, cross-shear, lateral splitting with end cone or wedge or some other combination of factors. Often, with weak mixtures there was no visible damage after maximum load had been ascertained by the drop of the beam.

No refined effort was made to detect actual closing up of cracks, but as far as noted, increased damage from retest included existing cracks. No cases were noted in which existing cracks did not seem to become an essential part of future damage.

Often moisture from within, first appeared at the surface at small cracks from the former test. Sometimes this squeezing out of moisture at retest gave the first visible indication of the very fine cracks from the former test.

These observations do not necessarily conflict with reports by others of cracks disappearing. As stated, no refined effort was made to check this phase of healing and the total period covered was possibly too short for complete closing of cracks under ordinary water storage. There were often evidences of soluble salt deposits near the surface at cracks.

Data on manner and extent of failure of individual specimens cannot be cited without making the paper longer than seems justified.

To save space a minimum of general and tabular data are included. Curves cover the tests rather fully and the treatment will consist of such explanations of curves as seem desirable and a few words at the end as to autogenous healing in its relation to engineering problems, practical and otherwise.

THE TESTS

The specimens were made and tests conducted in accordance with the best accepted procedure as followed at the University of Illinois, Lewis Institute and other leading laboratories with whose work and methods the author is thoroughly familiar. These methods are largely those described in the A.S.T.M. Standards. However, specimens were usually mixed several in a batch, but companion specimens came from different batches. In all cases ultimate strength was determined by the drop of the beam. Whenever there was any doubt as to ultimate strength having been attained, the loading was continued even though, in some cases, specimens were unnecessarily shattered thereby. Mean variation of individual strengths from the average strength of a companion group, at first test, was from 2.5 to 4.9 per cent for mortar 2 by 4-in. specimens and from 3.9 to 8.6 per cent for concrete (laboratory-made) specimens. There were rarely less than three specimens and in most series four specimens of a companion group. In retests the strength variation was great since some specimens of a group were bound to be more seriously damaged than others by the former test. Moreover, it was sometimes possible to salvage for retest all members of a group while in other cases, especially those of stronger mixtures and greater ages, none were saved. This accounts for gaps, in certain of the curves, such as show in B_2 and C_2 of Fig. 2.

The cement was of Colorado manufacture purchased in the open market. It passed the standard tests for portland cement.

In most series, the fine aggregate was a washed gravel 0 to No. 4 natural grading having a fineness modulus of from 2.5 to 3.0 in the different lots used. All in any one series was of the same lot and carefully pre-mixed to insure uniformity. Fine aggregates for different series came from different pits but were of the same general nature, free from organic material, well graded and with quartz grains predominating. Some few mortar tests, not shown, were made in which standard Ottawa sand was used. The healing occurred in the same manner and to the same approximate extent as with the natural sands of similar grading. The specific gravity of the natural sands used was 2.66.

The coarse aggregate used was a hard crushed sandstone (practically quartzite) of specific gravity 2.54 to 2.57. The grading differed for different series, but was in general either $\frac{1}{4}$ to 1 in. or $\frac{3}{4}$ to 1 in. Many tests of concrete made from this aggregate have shown it to

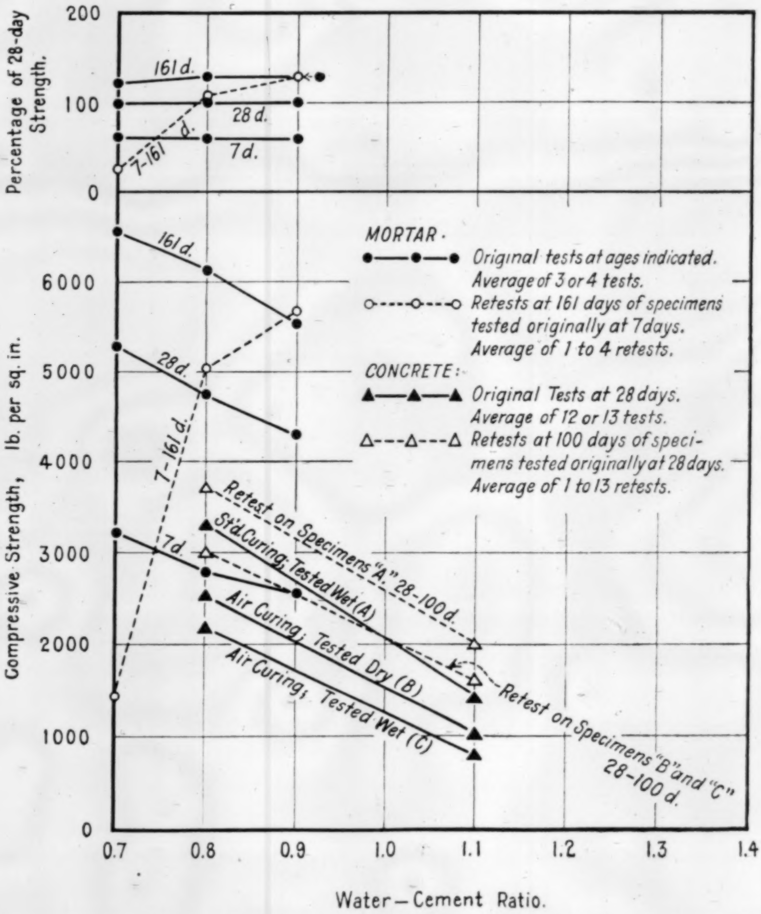

FIG. 1.—Relation Between Autogenous Healing and Water-Cement Ratio of Mortar and Concrete, Under Different Curing Conditions.

Mortar Specimens: 2 by 4-in. cylinders.

Aggregate, washed sand (0 to No. 4), specific gravity, 2.66; fineness modulus, 2.67.

Proportions 1:2.26 by loose volume (1:2.5 by weight).

Concrete Specimens: 6 by 12-in. cylinders.

Fine Aggregate, same as for mortars.

Coarse Aggregate, crushed sandstone (quartzite), grading $\frac{3}{4}$ to 1 in., specific gravity, 2.57; fineness modulus, 8.0.

Proportions 1:2:4 by loose volume (1:2.2:3.5 by weight).

All specimens were immersed in water after original test and not removed until retest.

be one of the best of the local materials. The fact that the maximum absorption is slightly under 1 per cent indicates that it is an unusually dense and impervious grade of sandstone.

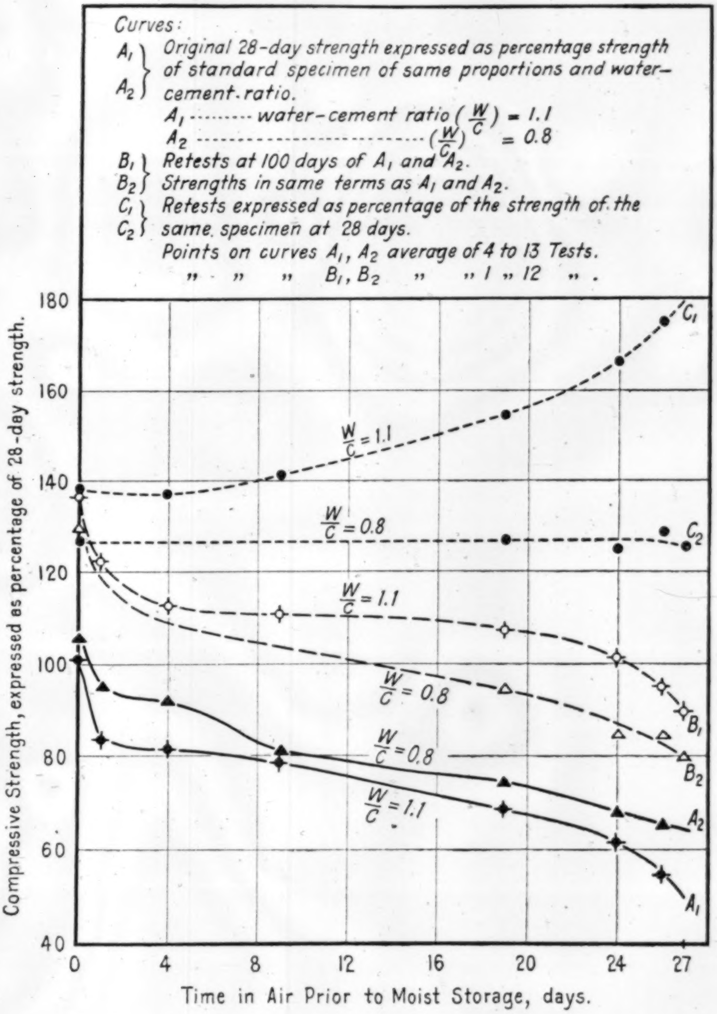

FIG. 2.—Relation Between Autogenous Healing of Concrete and Delayed Moist Storage During First 28 Days.

Specimens, 6 by 12-in. cylinders.

Proportions and aggregates same as for concretes of Fig. 1.

All specimens were removed from mold at age of one day and after varying periods of exposure to the air of the laboratory as indicated, were stored in water and tested wet.

All specimens were stored in water between original test and retest, and were wet at retest.

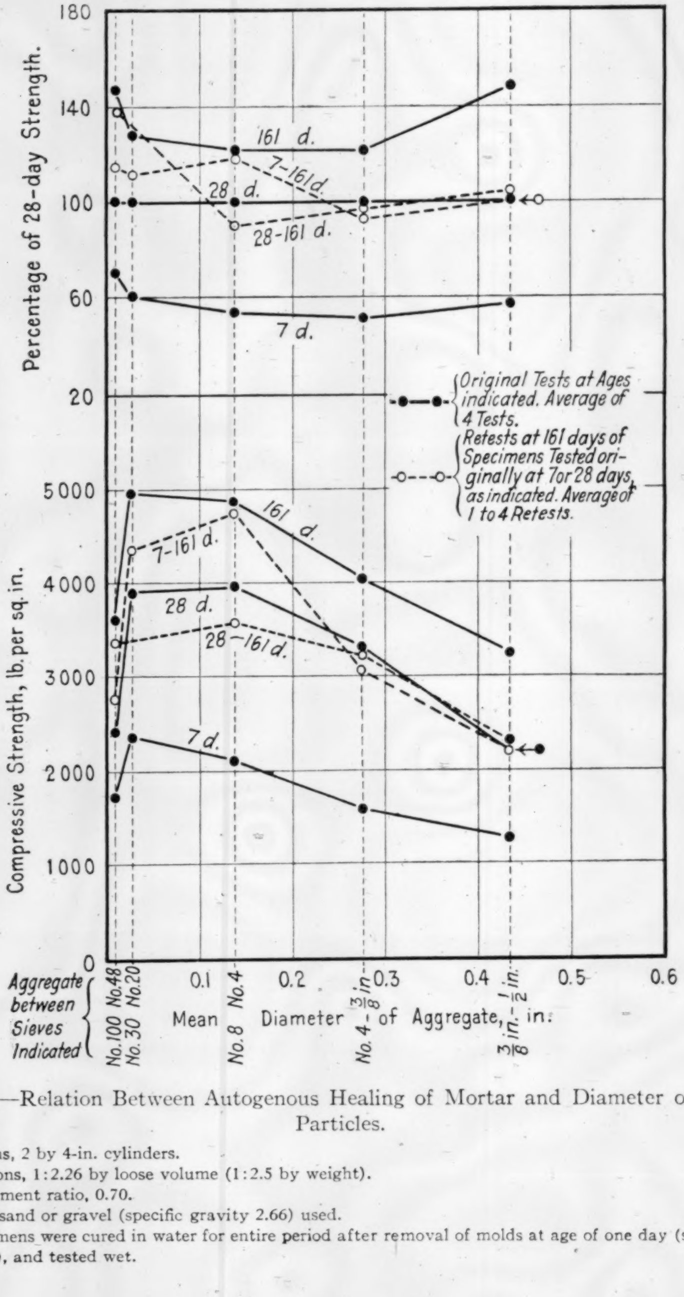

FIG. 3.—Relation Between Autogenous Healing of Mortar and Diameter of Sand Particles.

Specimens, 2 by 4-in. cylinders.

Proportions, 1:2.26 by loose volume (1:2.5 by weight).

Water-cement ratio, 0.70.

One-size sand or gravel (specific gravity 2.66) used.

All specimens were cured in water for entire period after removal of molds at age of one day (standard curing), and tested wet.

Moist storage was in all cases by immersion.

Figure 1 gives results of tests and retests of mortars and concretes of varying water-cement ratios. The retest of the mortars shows much greater strength gains for the higher water-cement ratios (lower strength mixtures). This probably does not indicate a fundamental difference in the extent of healing but rather a difference in the amount of initial damage. It was impossible to test the stronger mixtures to their ultimate strength without seriously damaging the specimens before the load could be released.

The concretes do not show such a marked difference between relative strength gains. The difference is present here too, however, as only a small part of the specimens having a water-cement ratio of 0.80 could be salvaged.

With equal initial damage, there is no evidence from the tests for believing that autogenous healing differs in either manner or degree in mortars and concretes or that water-cement ratio effects the relation except as it affects the initial damage.

The retests of concrete specimens show that the strength gain is more or less proportional to the initial strength. This is shown more fully in Fig. 2. Figure 2 shows results of tests of two concretes of 1:2:4 proportions of two water-cement ratios. Specimens were exposed to the dry air of the laboratory for various periods before being placed in water, after which all were initially tested wet at 28 days. They were then stored in water and retested at 100 days. The 28-day strengths varied widely due to the varied storage conditions.

The findings here agree with the tests of concrete given in Fig. 1, namely, that the strength of retested specimens is about proportional to the original strength. The results from specimens having a water-cement ratio of 0.8 are much less conclusive and satisfactory than those from specimens having a ratio of 1.1, because of the high degree of damage to the relatively few specimens of the first group that it was possible to salvage.

Figure 3 gives the results of tests and retests of mortar specimens, of 1:2.5 proportions (by weight) and water-cement ratio of 0.70, made with one-size sands or pebbles of varying diameter. The differences in strength for constant water-cement ratio are possibly traceable to the relative surfaces of contact between the aggregates and cement paste, strength being a matter of surface adhesion. It is interesting to note that gain in strength, both by added age and by autogenous healing, is practically proportional to the strength at first test. This statement does not hold for retests of mortar with aggregate of the two larger sizes used, which is doubtless due to the great damage

Curve	Symbol	Condition		Age, days		Material	Proportions		W/C	Std 28-day Strength, lb. per sq. in.	Storage Prior to First Test
		Test	Retest	Test	Retest		Loose Volume	Weight			
A	+	Dry	Wet	28	135	Mortar	1:2.26	1:2.5	0.7	4200	Wet-dry
B	○	"	"	28	100	Concrete	1:2:4	1:2.2:3.5	1.1	3303	" "
C	●	Wet	"	28	135	Mortar	1:2.26	1:2.5	0.7	4200	Dry-wet
D	▲	"	"	28	100	Concrete	1:2:4	1:2.2:3.5	0.8	3303	" "
E	△	"	"	28	100	"	1:2:4	1:2.2:3.5	1.1	1504	" "

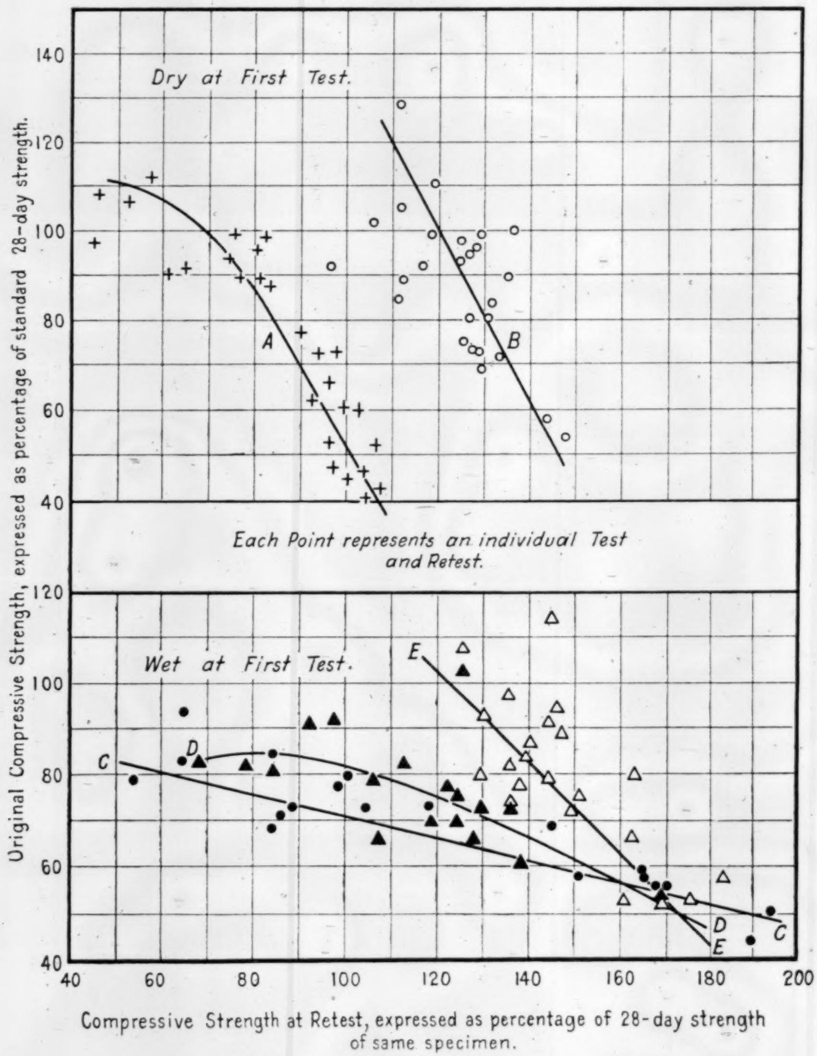

FIG. 4.—Relation Between Strength at Original Test and the Extent of Autogenous Healing.

Aggregates and proportions same as for mortars and concretes of Fig. 1.
All specimens were stored in water between original test and retest, and were wet at retest.

that is done to these specimens at initial test. With the larger pebbles in these cases, each stone is surrounded by a neat cement matrix which fails explosively at maximum load. The finer sizes of aggregate dilute the cement mortar sufficiently to make it less brittle.

Some tests were also made using standard Ottawa sand (20 – 30 mesh) as aggregate. These gave results similar to those shown in Fig. 3.

Figure 4 shows data for different concretes and mortars having a 28-day standard strength range of 1500 to 4200 lb. per sq. in., and an actual range, by virtue of varied curing conditions, of from 800 to 5000 lb. per sq. in. at 28 days. It illustrates clearly the percentage reduction in retest strength as the strength at original test increases. This is entirely ascribed to the more complete salvaging possible for weaker specimens. Curves *A* and *D* show a range over which the retest strength and original strength seem to bear little relation to one another. Above a certain strength the shattering effect is always sufficiently present to give a very low and erratic retest strength and the particular value of the latter is largely a matter of chance.

The extra steepness of curves *A* and *B*, representing specimens tested dry the first time, having been removed from moist storage at various ages prior to the 28-day test, is easily explained in part at least. By virtue of the hardening from drying out before the first test, the strengths are greater than they would be if the same specimens were tested in the saturated condition. This elevates all points. Then the same fact moves the point toward the left since the denominator of the horizontal scale is increased by the increased original dry-test strength. The varying slopes of curves *C*, *D*, and *E* are not so easily explained, but it will be noticed that curve *E* representing concrete of the highest water-cement ratio is steepest and that curve *C* representing the lowest water-cement ratio is flattest.

Several of the curves of Fig. 4 are not clearly defined, but this is to be expected because of the unavoidable high degree of variation in the extent of the damage from first test. The general trends do show, however, that strength at retest bears a marked relation to that at the original test. No essential differences between the relation for concretes and mortars are apparent.

Figure 5 shows the relation between time and strength of mortar for ages varying from two days to over eight months at test or retest. The mortar specimens were 2 by 4-in. cylinders, proportions of 1:2.5 by weight, with a water-cement ratio of 0.70 and a 28-day standard strength of 4556 lb. per sq. in. Numbers of tests as high as six are shown, that is, five retests. In general the maximum strength attained

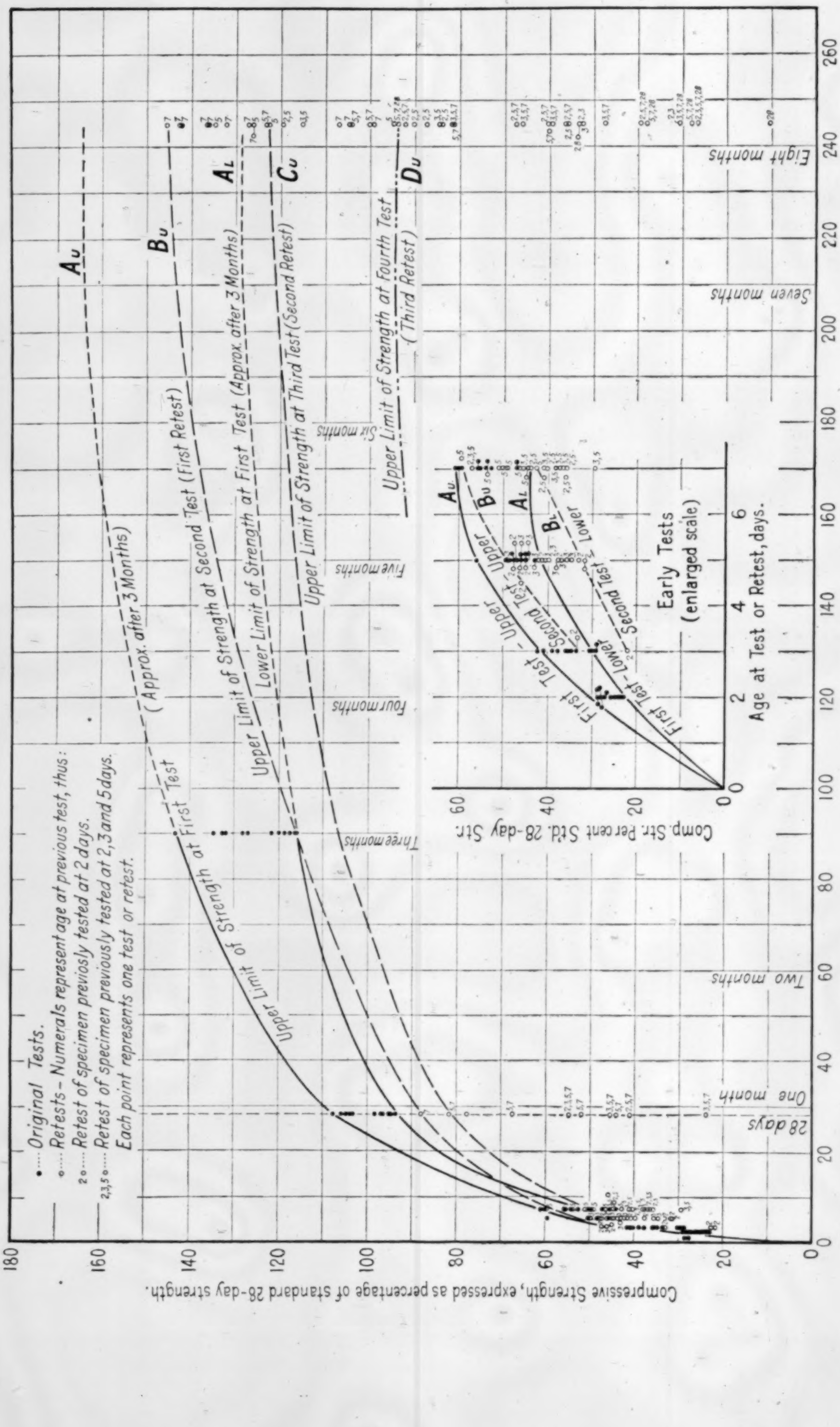

FIG. 5.—Relation Between Strength and Age of Mortars.

Original tests and varying number of retests of water-cured mortar specimens.
Mean 28-day strength, 4556 lb. per sq. in.
Specimens, 2 by 4-in. cylinders.
Proportions and aggregates same as for mortars of Fig. 1.

Water-cement ratio, 0.70.
All specimens were cured in water for entire period after removal of molds at age of one day (standard curing) and tested wet.

decreases at each age for each added retest. It must be remembered that this mortar was a strong rich mixture and the specimens were very brittle at ages above seven days. Leaner or weaker mixtures would doubtless have developed relatively higher retest strengths. The falling off of strength with each retest, in general, was doubtless due to added damage from the previous test. This curve is typical

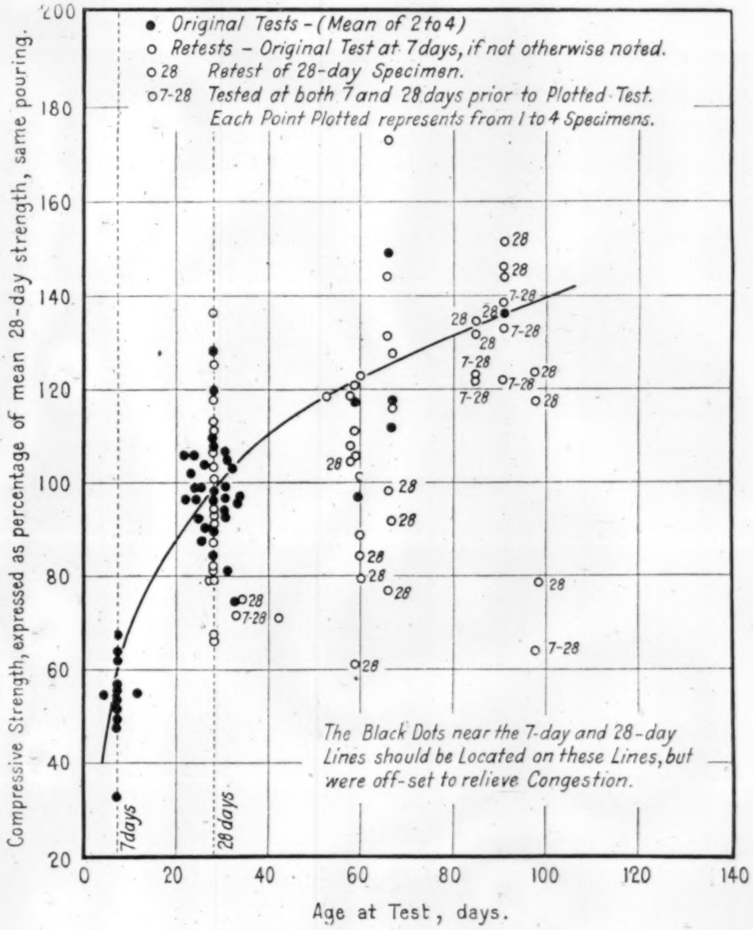

FIG. 6.—Original Tests and Retests of Concrete from a Typical Small Job.

Specimens, 6 by 12-in. cylinders. Slump ranged from 6 to 9 in.
Proportions variable.
Fine Aggregate, washed sand (0 to No. 4), natural grading, specific gravity, 2.65; fineness modulus, 2.75.
Coarse Aggregate, hard sandstone (quartzite), grading $\frac{1}{4}$ to 1 in., specific gravity, 2.54.
Original compressive strength at 28 days ranged from 1000 to 4000 lb. per sq. in.
All specimens were moist-cured and tested wet. All were wet between original test and retest.

of the rates of curing for concretes and mortars generally and forms a basis for interesting study.

Figure 6 shows the plotted results of some tests of job concrete, from a relatively small reinforced-concrete building project in which aggregates were proportioned by wheel-barrow loads and water determined by the "mixer-man judgment" method. The concrete was intended to develop from 2000 to 2500 lb. per sq. in. at 28 days. It actually did develop from 1000 to 4000 lb. per sq. in. The reinforcing bars were closely spaced and a very wet mixture was used (about 6 to 9-in. slump.) Seven and twenty-eight-day specimens were tested and the 7-day specimens were, in most instances, retested at 28 days. In general, each group of specimens, taken over about a half-hour period, agreed very well, but there was a rather wide range of strength from day to day. The retests averaged as strong as the original 28-day tests and the recovery from the 7-day test was evidently 100 per cent at 28 days. The black dots (original tests) near the 28-day period should all be located on the 28-day line but had to be off-set to relieve the congestion. The same is true for the 7-day group. Only a few original tests at ages above 28 days were made. One specimen in seven was generally saved for future tests.

GENERAL REMARKS

The extent of recovery in specimens badly shattered at former test is considerable. A typical case is that of a specimen tested at 7 days and so badly cracked that it could easily have been pulled apart with the fingers. It had after test a residual compressive strength of possibly one-tenth of its ultimate strength and practically no tensile resistance. The ultimate 7-day strength was 50 per cent of the 28-day strength of the "standard" specimen of similar proportions. At 28 days the specimen was retested and gave a new strength of 60 per cent of the 28-day "standard." This was a recovery of from about ten to sixty per cent. The specimens had about the same appearance before and after the 28-day test as just after the 7-day test, except for a rust-colored salt deposit near and in the cracks.

It is evident that any early cracking of job concrete, from premature removal of forms or other cause, may be almost if not completely healed, as regards ability to carry load (and possibly the cracks be made to disappear entirely, from Mr. Hollister's experience previously referred to) by replacing the props or braces and supplying a satisfactory curing environment for a week or more depending upon temperature, extent of damage and other conditions.

It may be of interest here to report that briquets of various proportions (including neat cement), that had been broken several months before, showed no signs of reuniting when the broken ends were held together for five days in water at 50° C. (122° F.). Recently tested compressive cylinders showed a slightly increased rate of healing when immersed in the same water for the same period of time. Freshly stored briquets often develop some little adhesion to one another when stacked for several days in the curing vats. This is probably an action similar to autogenous healing. More extensive tests along these lines would help to round out the subject.

Formerly there was ever present in the author's mind the question of whether or not a body of concrete was permanently injured if the forms were removed too soon and the concrete did not fail but was stressed almost to the point of failure. Such evidence as has here been considered seems rather conclusively to establish the fact that there is, during continued curing, a complete readjustment and annealing that excludes any thing resembling the initial stresses that may exist in a metal casting, for example. Probably shrinkage stresses due to drying out cannot be placed in this class because the concrete largely loses its capacity for internal readjustment when it becomes dried out.

The same applies to a test piece or beam in the laboratory, or to a pre-cast member on the job. If such a part falls or receives abnormally severe usage in handling, the ultimate strength is probably unaffected if it did not actually break and if the piece be in a moist environment for a short time after the jolt.

Some have used the principle of autogenous healing in removing hair cracks in decorative work. Possibly others can suggest additional applications of this very interesting property of concretes and mortars.

CONCLUSIONS

The following conclusions from the data presented seem justified in the way of general summary:

1. Autogenous healing takes place in much the same manner and under the same conditions as original curing. Concretes and mortars will not heal when dry (although there is a strength gain, in passing from the wet condition to the dry, which is merely a mechanical hardening, due to drying out).

2. All proportions and strengths of concrete or mortar will probably heal autogenously.

3. The extent of the healing (as compared with the strength

that would have been developed in the untested specimen) is mainly a function of the extent of structural damage at the former test or tests. Healing seems to occur in all cases, but the initial handicap is greater for specimens badly crushed, cracked, or shattered than for specimens less severely damaged.

4. Weak specimens (tested at early age, lean mixtures, high water-cement ratio, or poorly cured) give way slowly under load, and if the load is quickly removed the specimen may show little damage and may develop practically the same future strength as it would have developed had it not been previously tested. Strong mixtures usually crush, crack, or shatter at first test and, although future strengths may be greater than those at the time tested, they will rarely, if ever, approach that of undamaged concrete.

5. Recovery from damage at early age is more complete and rapid than that for old concrete. (The latter is much more brittle and in addition chemical processes are more dormant, just as strength gain in untested specimens continues at a constantly diminishing rate).

6. The number of former tests seems to have little bearing upon the healing, except in so far as more tests produce greater damage to repair. The same amount of damage at first test would probably have produced about the same ultimate result.

7. In the tests herein reported the evidence was of strength gain. Cracks did not in general disappear but there were deposits of salts in and around the cracks. Failure appeared usually, if not always, to be a continuation of that started by the former test whenever there was sufficient visible damage to define the nature of the former failure.

ACKNOWLEDGEMENTS

The tests were conducted in the concrete laboratory of the Department of Civil Engineering at the University of Colorado. Thanks are due to Prof. W. C. Huntington, head of the Department of Civil Engineering, for the manner in which he has encouraged the work of which these tests are a part. W. L. Eager, George M. Williams and George E. Breach, students in Civil Engineering, assisted in making tests and working up data.

DISCUSSION

MR. D. A. ABRAMS.[1]—Mr. Gilkey's tests on autogenous healing of concrete confirm a number of my past observations. This phenomenon was observed in tests of bond between concrete and steel bars made at the University of Illinois. **Mr. Abrams.**

Parallel tests were made on 1-in. plain round bars embedded axially in 8 by 8-in. cylinders. The specimens were stored in air or in damp sand. Two specimens for each condition of storage were tested at 5 days, 30 days, 3 months, and 1 year. The bars were pulled to the maximum load, corresponding to an end slip of about 0.01 in. At each test period, all specimens which had been tested previously were loaded to the maximum again; in this way some of the specimens were loaded to their maximum as many as four times (once at each of the above ages). The damp-cured specimens which had been previously loaded to a maximum once, twice, or three times, gave bond resistances equal to or greater than similar specimens which were loaded at the same age for the first time.

A complete report of these tests will be found in Table 21 and Fig. 43, *Bulletin No. 71*, University of Illinois Engineering Experiment Station, 1913.

MR. J. C. PEARSON.[2]—Recently our organization was building a walk to a new laboratory building, and in line with suggestions by Mr. Abrams on methods of getting higher strength concrete, we followed some of his recommendations in using a richer mix than usual, mixing for a somewhat longer time than usual, and using as stiff a consistency as was practicable under the conditions. The walk I have reference to was built in two days, and on both days we took samples of the concrete for test at 24 hours, 2 days, 4 days and 7 days. **Mr. Pearson.**

The strength of the concrete at 24 hours varied from 1500 to 2000 lb. per sq. in. and that at 7 days from 4300 to 5000 lb. per sq. in. Merely out of curiosity we removed the 24-hour specimens from the machine immediately after the ultimate load was reached and returned them to the damp closet for retest along with the 7-day specimens. The 7-day results of these retested cylinders were with one exception between 4300 and 5000 lb. per sq. in., although the average was slightly below the average of the normal 7-day specimens. Possibly

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[1] Portland Cement Association, Chicago, Ill.

[2] Assistant to Chemical Engineer, Lehigh Portland Cement Co., Allentown, Pa.

Mr. Pearson. a higher average might have been obtained with strict attention to the removal of the 24-hour specimens as soon as maximum load had been reached, but in any event the results are quite in line with those reported by Mr. Gilkey.

Mr. Richart. MR. F. E. RICHART.[1]—Recently at the University of Illinois we have been making a study of the failure of concrete under compression. In the tests that we made, the longitudinal and lateral deformations of the concrete were carefully measured up to the point of failure. At loads 75 to 85 per cent of the ultimate strength a decided change takes place in the volume of the concrete, which up to this point has been steadily decreasing. The lateral deformation now begins to increase proportionally much faster than the longitudinal deformation, so that before the ultimate strength is reached, the total volume of the cylinder has actually increased instead of having decreased. This increase in volume under compression seems to indicate a considerable internal rearrangement of the material in the specimen; perhaps some sliding, some splitting of the minute particles of the material. This abrupt change in volume is something that must be considered when we speak of autogenous healing taking place in a cylinder that has been carried to its ultimate strength. Although a cylinder may appear superficially to be undamaged after testing, there must still be a considerable amount of damage to repair within the material when autogenous healing is brought about.

Mr. Goldbeck. MR. A. T. GOLDBECK.[2]—The case mentioned by Mr. Richart is a very interesting one to me. Was the circumferential unit deformation enough to produce a tensile stress exceeding the tensile strength of the concrete?

Mr. Richart. MR. RICHART.—The lateral deformation certainly was very much larger than one would expect from our usual ideas of Poisson's ratio; That is, Poisson's ratio in this case was something over 0.5 at the untimate load and the lateral deformations were very large. It is evident that for some time before the ultimate load was reached, the lateral or circumferential unit deformation was considerably greater than the longitudinal unit deformation obtained at failure in a tension test.

Mr. Gilkey. MR. H. J. GILKEY (*author's closure by letter*).—The author appreciates the discussions offered. Those of Mr. Abrams and Mr. Pearson are similar to the general content of the paper and are very helpful in adding to its completeness. Mr. Richart's discussion gives us another angle of the subject.

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[1] Research Assistant Professor, Engineering Experiment Station, Laboratory of Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Director, Bureau of Engineering, National Crushed Stone Association, Washington, D. C.

Mr. Gilkey. As stated in the text, the paper purports to be only a brief record of observed phenomena. Some tentative explanations are offered but nothing that is intended to be considered as more than an opinion to be verified or discarded in the light of further knowledge. The eyes of expert investigators are now focused upon the chemical and physical chemical aspects of cement and concrete, as well as upon the purely physical phenomena such as those here recorded. There is no question but that the near future will see these different manifestations fully coordinated and adequately explained. Such information as that contributed by Mr. Richart is one more step toward that end.

The fact that specimens do recover strength in spite of the very serious internal disturbance, should be an added stimulus to these qualified investigators.

A SIMPLE DEVICE TO OBVIATE CAPPING OF CONCRETE SPECIMENS

By Wallace F. Purrington[1] and James McCormick[2]

Synopsis

This paper describes a device for use in the compression testing of concrete specimens, consisting of a container retaining a sand cushion in contact with the specimen. The usual method of capping is thus made unnecessary. Test results are given, showing the variations to be expected in this method of testing as compared with the standard method.

Probably the greatest problem connected with the testing of concrete field samples lies in the fact that it is very difficult to have the average foreman or inspector take the trouble necessary to properly cap the cylinders, following the method as outlined in the A.S.T.M. Standard Methods of Making and Storing Specimens of Concrete in the Field (C 31 - 21).[3] In the field we have found it more satisfactory to use paper molds. With these molds it is very difficult to use a glass or metal plate to work the neat cement down to a satisfactory bearing surface unless the greatest care is taken, since the top surface is far from being a smooth plane.

The time between the making of the concrete sample and the time of capping should be such that both the sample and the neat cement would be properly shrunk. If the samples were made up in the morning this would cause no great difficulty, but if these samples were formed in the late afternoon it would be necessary for someone to be on the job at the proper time to cap these cylinders, which means that this person must remain after the day's work is completed.

We are using a device which is simple, inexpensive and satisfactory to eliminate capping and which at the same time will give us an even bearing surface. When sand is confined, an excellent bearing is provided. The principle involved is similar to that used in the sand-bearing test for pipe. Figure 1 shows the construction and manner of placing the apparatus.

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[1] Materials Engineer, New Hampshire Highway Department, Concord, N. H.

[2] Assistant Materials Engineer, New Hampshire Highway Department, Concord, N. H.

[3] 1924 Book of A.S.T.M. Standards.

To determine the accuracy of the method a series of twenty-four 6 by 12-in. cylinders were built capping these by the standard method, and the same number were formed which were not capped. The tops were simply troweled after filling the cylinders and kept covered with oiled paper. Of these only one broke which gave a result of more than fifteen per cent from the average.

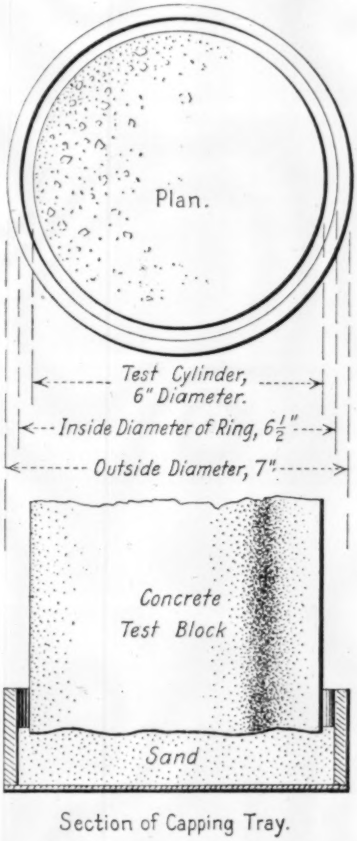
Plan.
Test Cylinder, 6" Diameter.
Inside Diameter of Ring, 6½"
Outside Diameter, 7"
Concrete Test Block
Sand
Section of Capping Tray.

FIG. 1.—Showing Concrete Test Specimen in Position.

The following tabulations show the individual breaks of the samples tested. In Series A and B, the mix was 1:2.98:3.14, the aggregate being Topsfield sand and gravel. The amount of water used was 6.75 gal. per bag of cement. The mix was calculated to give a strength of 2000 lb. per sq. in.

Sample	Series A Compressive Strength, lb. per sq. in		Series B Compressive Strength, lb. per sq. in	
	Capped	Uncapped	Capped	Uncapped
No. 1	2056	1872	2231	2440
No. 2	2167	2105	2132	2227
No. 3	2013	2160	2289	2003
No. 4	2029	2052	2388	2006
No. 5	2047	2161	2353	2101
No. 6	2175	....	2300	2164
Average	2061	2070	2282	2157
Maximum	2175	2161	2388	2440
Minimum	2029	1872	2132	2003
Variation between maximum and minimum	146	299	256	447

In Series C the mix was 1:2.9:3.1 and in Series D 1:2.66:2.84. In both Series C and D the aggregate was Topsfield sand and Winchester trap rock, and 5.25 gal. of water were used per 1 bag of cement. The mix was calculated to give a strength of 3000 lb. per sq. in.

Sample	Series C Compressive Strength, lb. per sq. in.		Series D Compressive Strength, lb. per sq. in.	
	Capped	Uncapped	Capped	Uncapped
No. 1	3044	2747	3077	3043
No. 2	3195	2558	2610	2787
No. 3	2949	2961	3084	3185
No. 4	3223	2850	3244	2912
No. 5	3507	2812	3063	3000
No. 6	2499	2710	3327	2546
Average	3070	2773	3068	2912
Maximum	3223	2961	3327	3185
Minimum	2499	2558	2610	2546
Variation between maximum and minimum	724	403	717	649

CONCLUSIONS

For field samples, it is believed the capping device will give more accurate results than will cylinders capped by the standard method by persons without laboratory experience. From the above tests it can be seen that either method gives breaks which are within the limits usually used for this type of work.

DISCUSSION

Mr. Ellis.

MR. A. R. ELLIS.[1]—I should like to ask Mr. Purrington if there is any relation between depth of the sand cushion for different kinds of tests, and also if there is any special kind of sand to be used in the cushion.

Mr. Purrington.

MR. W. F. PURRINGTON.—I will answer the last question first. What we use is a sand of about between 20 and 30 mesh, but after we have used the sand a couple of times it grinds down to a fine flour, whatever its size. After a while this material gets a little hard, and we break up the crust with our fingers and smooth it out roughly or shake it just to get a level bearing. We do not take any special account of the size of the sand.

Now, so far as the depth of the sand cushion is concerned, we started in with simply an iron ring that was put in a pan, and was filled half or two-thirds full. In these tests there were two or three different lots of sand used. We tried to keep thoroughly fresh sand and we took no account of the depth, simply using enough so that the bottom of the cylinder did not by any possible chance touch the bottom of the capping device.

Mr. Goldbeck.

MR. A. T. GOLDBECK.[2]—I should like to ask whether there is a rubber gasket to confine the sand between the iron ring and the cylinders.

Mr. Purrington.

MR. PURRINGTON.—No, we used nothing. The ring was just large enough to get the cylinder in with about $\frac{1}{4}$ in. clearance. We do not get any field specimens exactly 6 in., using paper molds; they will vary somewhat. We simply wanted just about the least space we could have between the cylinder and the ring.

Mr. Gonnerman.

MR. H. F. GONNERMAN.[3]—I should like to ask Mr. Purrington if he has made any tests with the sand cushion using concrete stronger than 3000 lb. per sq. in. at 28 days. The tests for 3000-lb. concrete seemed to show a decrease in strength of about 10 per cent with the sand cushion and I was wondering if a greater decrease might not have been obtained if concrete having a strength of say 4000 or 5000 lb. per sq. in. at 28 days had been tested.

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[1] General Manager, Pittsburgh Testing Laboratory, Pittsburgh, Pa.

[2] Director, Bureau of Engineering, National Crushed Stone Association, Washington, D. C.

[3] Associate Engineer, Research Laboratory, Portland Cement Association, Chicago, Ill.

Mr. Gonnerman. Tests of 6 by 12-in. concrete cylinders were made at the Structural Materials Research Laboratory[1] using a variety of sheet materials, which included beaver board, white pine, leather, lead, etc., between the top of the cylinder and the bearing block as a substitute for neat cement capping; the top of the cylinder was troweled to a smooth surface at the time of making and the bottom molded to a true plane by use of a machined plate under the form. The difference in strength of the cylinders tested with the sheet materials compared with that of cylinders capped in the standard manner with neat cement was greater the stronger the concrete, although the mean variations in the tests were generally about the same. The cause of the lower strength was a splitting action induced by the tendency of the sheet material to flow laterally when under load, this splitting action apparently being more pronounced the stronger the concrete.

Mr. Purrington. MR. PURRINGTON.—No, we have not tested the higher strength concrete in this way.

Mr. Proudley. MR. C. E. PROUDLEY.[2]—I should like to ask if a portland-cement cap was used, and if so, what was done to insure a plane surface.

Mr. Purrington. MR. PURRINGTON.—The caps were of neat cement and made up three or four hours prior to capping. We brought the cylinders down to a bearing with the glass plate and a piece of oiled paper, and we were very careful to get as near a perfect surface as we possibly could. Then we would test every cylinder with a straight edge before it was broken, to see that it was reasonably smooth. I should not want to say that this surface was absolutely perfect, but what one would get practically in finishing a cylinder.

Mr. Smith. MR. J. HAMMOND SMITH.[3]—I am very glad that this method of testing concrete cylinders has been brought out before the Society in the form of a paper based on comparative tests. We have been using this method at the University of Pittsburgh for several years with excellent results. Our method is simply to take a 6-in. pipe cap and plane it off on the bottom, or outside of the closed end. This is used on the bed of the testing machine with no further finishing whatever. About 1 in. of 20-mesh sand in the cap as a bed for the cylinder works very well. It is necessary, however, to stir the sand up, renew or sieve it as occasion demands, as broken pieces from a test cylinder may interfere with the proper bedding of the next cylinder.

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[1] H. F. Gonnerman, "Effect of End Condition of Cylinder in Compression Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 1036 (1924).

[2] Assistant Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[3] Professor of Civil Engineering, University of Pittsburgh, Pittsburgh, Pa.

SYMPOSIUM ON ROSIN

WHY A ROSIN SYMPOSIUM?

Introduction by F. P. Veitch[1]

Rosin has long been bought and sold on the basis of its color and cleanliness alone, without any consideration of its chemical or its other physical properties. During the past eighty years a number of "grades" as indicated by color have been established, and types for these recognized first by the trade, then by the laws of Georgia and Florida, and finally on March 3, 1923, by the Federal Naval Stores Act, which made "The rosin types heretofore prepared and recommended under existing laws, by or under authority of the Secretary of Agriculture" the standards for rosin until otherwise prescribed as thereinafter provided. These "types" for color prepared from colored glass by the Department of Agriculture were, as a matter of fact, made to duplicate the average type samples of rosin long used in the industry.

With the development of new uses for rosin, and a better understanding and closer control of processes in which rosin serves as a raw material, there has developed the recognition of a need for more knowledge of all the chemical and physical properties of rosin, and of how these can be modified and determined in order that rosins may be used more intelligently, more effectively, more economically and perhaps more largely in the numerous uses to which they are put.

While the chief constituents of rosin are known in a general way, it cannot be said that we know all of them, or whether or not these constituents, or the so-called constants of rosin, are the same or essentially the same for all grades of rosin; nor do we know how, through producing processes, to modify or change the proportion of the several constituents or to control the constants. The methods for examining or technically evaluating rosin are, to say the least, not standardized, and much remains to be done in the way of modifying rosin to make it more suitable for a given use or to adapt it to new uses.

As one of the first steps in making known to producers the needs of the users of rosin, of acquainting the users with the existing conditions of production, and to lay a better foundation for its future work,

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[1] Chemist in Charge, Leather and Paper Laboratory, U. S. Bureau of Chemistry, Washington, D. C., and Chairman of Committee D-17 on Naval Stores of the American Society for Testing Materials.

Committee D-17 on Naval Stores decided at its last meeting in Cleveland, October, 1925, to hold this Symposium on Rosin.

The papers that will be presented are intentionally brief. They do not attempt to utter the final word on any subject but each will serve to introduce the subject; and on it and the subsequent discussion that it is hoped will be full and informing, it is confidently expected that it will be possible to base effective work by the committee and to help develop the more intelligent production and use of rosin.

ROSIN FOR VARNISHES

By E. W. Fasig[1]

Rosin, when compared with the fossil resins or copals, is an inferior resin for varnish making purposes, in some respects, but it plays an important part in the industry and some excellent varnishes are made from it.

In the privacy of his own plant, the varnish maker incorporates this resin into his products, feeling full well that he is producing materials that will satisfactorily meet given conditions; yet to the public he is inclined to disclaim any knowledge of the use of rosin and with profound piousness maintains his products are free from such a disgraceful component. It is true that some very bad varnishes have been made from rosin, but with rosin, as with most other materials, new methods of treatment have been developed and to-day we find some very high-grade durable varnishes the gum base of which is prepared from it.

It is common practice to make varnishes from both untreated and hardened rosins. The rosin may be dissolved in volatile solvents with or without the addition of drying oils. The solution may be made by the aid of heat, or may be effected in the cold.

Hardened Rosin.—There are a number of ways in which rosin is hardened, the most common method being to treat it with lime or zinc oxide. Lime of a high CaO content is used and the addition of 5 or 6 per cent neutralizes a part of the acids of the rosin and results in a harder product than the original. The lime is dusted into the melted gum and a rather rapid combination results. Zinc oxide may be substituted for the lime, if preferred. The procedure is the same in both cases.

Rosin Ester.—Another way of preparing rosin for varnish making purposes is to combine it with glycerin. By this method, the acids of the rosin are converted into glycerides, the acid value of the original rosin being reduced from around 155 to 8 or 9, or even lower. This ester is a very desirable base for high-grade varnishes; when combined with tung oil, varnishes possessing high water and weather-resisting qualities are obtained. Such varnishes need no apologies from the producers and the silly explanations and excuses for making such products should be discarded. Such practices play to public ignorance. It is time to educate the public to the fact that rosin can be prepared in such a way that high-grade varnishes can be made from it.

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[1] Assistant General Superintendent, The Lowe Brothers Co., Dayton, Ohio.

Other Metallic Resinates.—In general, three metals are used as accelerators in the drying of varnishes: lead, cobalt and manganese. It is quite common to add these metals in the form of resinates. There are advantages and disadvantages in these compounds over the corresponding metal compounds of vegetable oils, depending upon the conditions under which they are used. It is the author's opinion that these metal resinates, contrary to the somewhat popular belief, affect the durability of varnishes little differently than do the linoleates.

Rosin as a Flux.—It is quite a general practice to use rosin in the form of a flux for other varnish resins. The acid of rosin seems to be an efficient dispersion agent or in some cases a good solvent, when mixed with other resins. This property assists in the solution of these resins, thereby eliminating some of the loss due to destructive distillation.

PROPERTIES OF ROSIN

Unwilling to rely entirely upon my own judgment as to the desirable properties of rosin for varnish making, I sent a questionnaire on the subject to fourteen of my acquaintances in different parts of the country. I received replies from eleven of them. Before recording the questions and the replies, I should like to quote as follows from a letter by Mr. Carl Bragdon in reply to the questionnaire:

> "Before answering the questions in detail, we call your attention to the fact that rosin as used in varnish making is cooked at temperatures of 500 to 575° F. (260 to 302° C.), and that the properties desired in the finished varnish are really those of the rosin when it has been through the heating process, which may differ from the rosin as it is originally received. Thus, if the rosin in its original distillation has not been carried far enough to remove all of the volatile oleoresins, it will be softer and have different acidity, etc., than after it has been cooked in making varnish from it. Inasmuch as there have been no standards or specifications for rosin other than color, and in view of these changes in properties on cooking, we have paid little attention to chemical and physical tests of our shipments."

Questionnaire:

1. *Is the melting point of rosin important to you?*
   Eight answered yes; three no.
2. *Do you determine the melting point?*
   Three answered yes; eight no.
3. *Do you determine the amount of dirt in the rosin you buy?*
   One answered yes; ten no.
4. *Assuming that the rosin contains no unusual amount of dirt, is a dirt determination of any great value in judging the quality of the rosin for your purposes?*
   Eleven answered no.

5. *How do you check the color?*
   Six replied that they check the color against U. S. Government standards; five use their own standards.
6. *Do you consider the acid value of the rosin important?*
   Eight considered it important; three did not.
7. *Do you consider the saponification value of any great value as an aid in judging the quality of rosin?*
   One answered yes; ten no.
8. *Is the iodine number of any value?*
   One answered yes; nine no; one thought it of doubtful value.
9. *Do you consider the softening point of any great importance?*
   Six answered yes; five no.
10. *Is hardness of any importance to you in judging the quality of the rosin?*
    Eight answered yes; three no.
11. *State your preference as to the kind of package, metal or wood?*
    Nine preferred metal; one preferred wood; one had no preference.

Judging from the replies to the questionnaire, there seems to be quite a wide difference of opinion. Practically all are agreed that the average amount of dirt found in rosin is not greatly objectionable. To most, the melting point is important, but there seems to be no satisfactory method of determining this. All have methods of judging color. Those who manufacture rosin ester are interested in both acid and saponification values. The iodine value seems of little importance. Most varnish makers prepare a small batch of varnish from a representative sample of a shipment of rosin and from this varnish they judge the hardness as well as other properties. Those who replied to the questions are almost unanimous in their choice of metal containers. They, however, point out that any iron rust is highly objectionable.

CONCLUSION

The most important thing so far as the varnish maker is concerned is to obtain a rosin which, when incorporated in a varnish, will meet all established conditions for that varnish. The general practice has been to make a small batch of the varnish from a representative sample of the shipment of rosin and thereby judge the quality.

The varnish industry has experienced trouble at times with what seems to be a crystallizing out of part of the rosin from solution. This produces a "seedy" effect when the varnish dries, and is very objectionable. It is evidently a crystallin condition of rosin which is not as soluble as the other or regular material.

DISCUSSION

Mr. Veitch. MR. F. P. VEITCH.—I should like to ask what percentage of dirt Mr. Fasig thinks would affect the value of rosin for varnish making; or if he has any results on dirt determination.

Mr. Fasig. MR. E. W. FASIG.—The only dirt determinations we have ever made, to my knowledge, were made on the samples of rosin you sent us. We have paid very little attention to dirt in rosin, for the reason that the dirt usually found there is of the kind that does not affect the color of the final product and can be eliminated in the final product by filtration.

Mr. Walker. MR. P. H. WALKER.[1]—I am rather surprised at Mr. Fasig's insistence upon the importance of the color of rosin. Apparently no one uses rosin in its original condition and I should think that if color is an important item, it would be the color of rosin after it has been treated by heat, as is necessary in the various uses of rosin.

Mr. Fasig. MR. FASIG.—I agree with Mr. Walker in what he has said, but we have found too that both esters and varnishes made from the paler grades of rosin are paler in color than those made from the darker grades of rosin.

Mr. Kinney. MR. C. E. KINNEY.[2]—I wish to ask Mr. Fasig whether unsaponifiable matter has a detrimental effect on esterification.

Mr. Fasig. MR. FASIG.—I do not know what effect the unsaponifiable matter has on esterification, for we have never paid any attention to it in the manufacture of our esters. We determine the acid value, and from this figure we make our batch formulas.

Mr. Romaine. MR. E. V. ROMAINE.[3]—Is the reaction in forming the ester gum dependent upon the acid value or the saponification value of the gum? There is a difference.

Mr. Fasig. MR. FASIG.—I am not prepared to say whether or not the reaction in forming ester gum is dependent upon the acid value or saponification value of the gum. We are quite positive, however, that the acid value is a true indication of the amount of glycerin to be used, for by using this figure we are able to control the acid value of the final product very closely.

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[1] Chemist, U. S. Bureau of Standards, Washington, D. C.

[2] Manager, Technical Service, Naval Stores Division, Hercules Powder Co., Wilmington, Del.

[3] General Naval Stores Co., New York City.

Mr. Kesler.

MR. C. C. KESLER.[1]—The question was asked a few moments ago as to what affect the unsaponifiable material had upon the esterification. From preliminary tests, I should say it has a marked retarding effect upon the ease with which it is esterified.

Mr. Grotlisch.

MR. V. E. GROTLISCH.[2]—Several years ago it was brought to the attention of the Bureau of Chemistry that one of the prominent manufacturers of rosin ester received a lot of rosin which turned out an ester of the same color as if he were using "F" rosin. I wonder if Mr. Fasig has had that experience, having water white rosin give a dark colored ester. This particular rosin comes from one particular locality in the South. We have tried to get more light on it and have been unable to do so.

Mr. Wertz.

MR. F. A. WERTZ.[3]—There is one kind of rosin the varnish makers do not want—at least we do not want it—and that is opaque rosin. We have had some barrels a very high percentage of which was quite opaque, rather the color of coffee with cream in it. I think the opacity is entirely due to moisture and it was the cause of a very nice little fire in our place.

Mr. Brown.

MR. B. S. BROWN.[4]—I should like to ask the gentleman how long it has been since he has had opaque rosin.

Mr. Wertz.

MR. WERTZ.—About three weeks.

Mr. Brown.

MR. BROWN.—Opaque rosin is an unmerchantable product according to the Naval Stores Section, Board of Trade, and you have a right to object to anything like that.

Mr. Grotlisch.

MR. GROTLISCH.—My understanding is that opaque rosin is merchantable but must be sold as opaque. It has to be sold on samples for what it is. There is opaque rosin which is caused by crystallization, or the so-called sugaring out of the rosin. Does that work up into a varnish readily—opaque rosin due to crystallization but with no water present at all? It is harder than ordinary rosin. Could it not be made into a harder varnish?

Mr. Fasig.

MR. FASIG.—The opaque rosin of which Mr. Grotlisch speaks is what I referred to in the final paragraph of my paper. This crystallization causes a great deal of trouble in varnishes and enamels.

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[1] Mellon Institute, Pittsburgh, Pa.

[2] Bureau of Chemistry, Washington, D. C.

[3] Production Manager, Varcraft Works, Inc., Pottstown, Pa.

[4] President, Georgia Rosin Products Co., Brunswick, Ga.

THE USE OF ROSIN IN INSULATING AND POTTING COMPOUNDS AND IN SEALING WAXES

BY F. L. ROMAN[1]

The insulating and potting compounds which are here referred to are used mainly by the telephone, radio and other electrical industries, while the sealing waxes are used more generally for purposes which do not involve electrical insulation. There is no sharp distinction, however, between insulating compounds, potting compounds and sealing waxes, the nomenclature referring to the different purposes for which the products are used rather than to different types of material. The same compound may be used in some cases, for example, as an insulating compound and as a potting compound. The insulating compounds are used mainly to impregnate paper, textiles and similar materials used in condensers, on wire and coils, and for miscellaneous parts requiring electrical insulation. Many of these types of condensers, coils and other parts are then placed in cans or cases which are filled with so-called potting compounds. These potting compounds are intended in general to provide electrical insulation and in most cases to hold the part in the desired position and to protect it from mechanical injury.

Rosin enters into many insulating and potting compounds, mainly because it has good electrical insulating properties. Its stickiness, ready miscibility, high melting point and low cost as compared with many gums and waxes, also make its use desirable in such compounds. Its brittleness, however, limits the quantity of rosin which may be used in some of these products.

Two grades, WW and F, are generally used. The use of light-colored rosin is required for light-colored compounds, such as are used, for example, on the textile insulation of certain types of wire. Colored compounds would not only yield unsightly wires, but would make it difficult to distinguish the wires of different colors and to make proper connections. Grade F rosin, either wood or gum, is commonly used for making compounds, because it is clear, reasonably free of impurities and readily available. A darker rosin may be used in a few cases.

The rosin is used as follows:

1. As received, in potting some types of coils (filling the case around the coil);

2. Mixed with various proportions of rosin oil, the mixture being used to impregnate the textile insulation of certain coils;

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[1] Chemical Engineer, Western Electric Co., Chicago, Ill.

3. Hardened with various proportions of hydrated lime. Hardened rosin may be used in potting certain types of coils in place of rosin. Its advantage is that because of its higher melting point it is less apt to flow out of the case if the coil heats up under service conditions. The hardened rosin also enters into a number of compounds in which a product of higher melting point than rosin is required;

4. Incorporated into a variety of compounds, for example with asphalt and rosin oil, with beeswax and fillers, with paraffin, with carnauba wax and beeswax, etc.

While it is desirable that the rosin used for the above purposes be reasonably clean, the difficulties due to impurities in the rosin are comparatively small. Troubles from moisture and from variations in melting point are by far the greatest. Moisture even in very small quantities causes radical reductions in the insulating properties of the rosin and compounds. A small amount of this moisture may be present in the rosin itself as opaque rosin, but most of the moisture which enters in the compounds is due to water carried along mechanically in depressions and fissures in the rosin. The difficulties from this source have been such that the rosin is in many cases stored indoors for several weeks before it is used, and in some instances the rosin is broken up in small pieces and spread out indoors for a few days before it is used.

In order that they will remain satisfactory under service conditions, the melting point of many compounds must be maintained above a certain point, and the variations in the melting point of rosin have been the cause of some trouble. The main difficulty from this source occurs, however, in compounds using hardened rosin. Even with rosins of the same melting point, two different lots are apt to give, under closely controlled methods of manufacture, hardened rosins showing considerable variations in melting point. Although this has not been shown definitely, there are indications that gum rosin yields hardened rosin of higher melting point than does wood rosin.

The acid number, saponification number and iodine number of the rosin do not appear to give any indication of the value of the material for use in insulating and potting compounds.

Rosin enters also in most sealing waxes (usually in combination with shellac, turpentine, fillers and coloring material) and in similar compounds which need not possess good dielectric properties. A clean rosin of reasonably uniform melting point is desirable, but the presence of traces of moisture is not so objectionable as in the case of the insulating and potting compounds.

DISCUSSION

Mr. Lockwood.

MR. J. E. LOCKWOOD.[1]—At one of our previous meetings I believe Mr. Roman called attention to the fact that the trouble due to moisture in insulating compounds has proved to be serious even when the amount was very low. I believe he said that even as low possibly as 0.05 per cent of moisture has proved serious. I think, as we now have a very large and representative attendance, if he could tell us a little more clearly what the problems are and what information he needs or what help can possibly be given by the producer of rosin to eliminate his trouble or to show if it is caused by something other than rosin, it may be we can get some more light on the subject.

Mr. Roman.

MR. F. L. ROMAN.—The question of the troubles caused by moisture in rosin used for insulating compounds was brought up in the discussion of the methods of determining moisture in rosin. I believe the method of determining moisture by means of xylol was discussed. So far as I know this method will not give results which are accurate to more than 0.1 or 0.2 per cent. I pointed out that it would be desirable to obtain a method which would give results much more accurate, probably more accurate than 0.05 per cent.

Rosin is used in compounds which may be used in large cases. Certain types of cases may hold as much as a thousand pounds or more of compound. As little as 0.05 per cent of moisture in such a product would amount to about half a pound of water in that case. Assuming that the compound were being poured hot in the case and that moisture had an opportunity to accumulate in a cold portion of the container, it might lead to a great deal of trouble from an electrical standpoint. From our standpoint it would be desirable that a method be worked out which gave results more accurate than the xylol method.

Mr. Hanson.

MR. C. F. HANSON.[2]—Inasmuch as Mr. Veitch has asked us to tell about our troubles, I might mention a few that I have had. I am interested in the manufacture of underground power cables. These consist mainly of a copper conductor wound with paper which is subsequently dried by a vacuum process and then impregnated. Up to four or five years ago it was our practice to use as an impregnating compound a mineral base oil containing a certain percentage of rosin.

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[1] Manager, Naval Stores Division, Hercules Powder Co., Wilmington, Del.

[2] Habirshaw Cable and Wire Corporation, Yonkers, N. Y.

Mr. Hanson. We found, however, that with that type of compound we were unable to obtain the low power factor which our trade demands. Rather than the term "power factor," which is difficult to define, I shall use the term "resistivity." Later, I shall explain the relation existing between power factor and resistivity.

The unit of resistivity, ohm-centimeter, is the resistance offered by a cubic centimeter of compound when the resistance is measured between two opposite faces of that cube at 85° C.

Here is an interesting phenomenon. Consider a mineral base oil with a fairly high resistivity, for example 10×10^{12} ohm-centimeters at 85° C. Now, if I take rosin which has a resistivity as high as 100×10^{12} ohm-centimeters at 85° C. and mix 15 or 20 per cent of it by weight with the oil, the resistivity of the oil is reduced quite considerably below its original value.

This reduction in resistivity would not be so bad if that were the whole story. When this mixture is used in our impregnating tanks, although a vacuum process is used, it is almost physically impossible to keep that compound away from air at all times. It will become exposed to air at certain times, particularly due to the small leaks at the bottom of the tank. This leak of air which rises up through the mass of impregnating compound will oxidize the compound. Some may take issue with me for using this term "oxidize" but according to the best information that the oil chemists can give me it is an oxidation process. As mentioned, this oxidation will take place and as the compound becomes oxidized its resistivity falls off quite rapidly. This oxidation is rather curious in one respect, because ordinarily the mineral oil containing the rosin will oxidize much more rapidly than the oil alone. That is rather curious for this reason: Rosin oil, which is a product of rosin, when blown with air, will increase in resistivity. At first hand it would seem that here would be a fortunate combination of two materials. The tendency of the rosin to increase in resistivity should neutralize the tendency of the mineral oil to decrease in resistivity when the mixture is exposed to air at high temperatures.

In my experience, this neutralizing effect of the rosin does not occur, but on the contrary, the rosin acts as a catalyzer to oxidation.

In closing, I wish to call your attention to the relation between resistivity and power factor, which relation, according to my experience, holds good for mineral base oils and mineral oils mixed with rosin, and which I believe applies with respect to rosin oil. As resistivity decreases, the power factor increases and cables are sold on the merits of low power factor.

Mr. Hanson. The most interesting phase of this whole thing is: What happens in the chemical structure of the oil during oxidation? This question I cannot answer. It is a very vital question in the insulation industry and becomes more vital as we increase the operating voltage of our cables. Until a few years ago, 13,000 volts was about the normal operating voltage, but now we are going up to 132,000 volts, and as we increase the operating voltage this problem becomes more and more acute.

Mr. Grotlisch. Mr. V. E. Grotlisch.—May I ask at what temperature the mass is run in which you get the oxidation?

Mr. Hanson. Mr. Hanson.—Anywhere from 80 to 150° C. The higher the temperature, the greater the rate of oxidation.

Mr. Grotlisch. Mr. Grotlisch.—It has been brought out that rosin oxidizes very readily when melted and one of the products of oxidation is water. Probably the formation of water is the reason for loss of resistivity. If the tank, or whatever you use for cooking the batch, could be insulated with a non-oxidizing gas like carbon dioxide, you might get around the trouble.

Mr. Walker. Mr. P. H. Walker.—I doubt if that can be done. There was an article in the June, 1926, number of *Industrial and Engineering Chemistry* in which the authors (Shaw and Sebrell) show that commercial carbon dioxide contains sufficient oxygen to oxidize rosin.

Mr. Grotlisch. Mr. Grotlisch.—It should be better, at least, than passing air through. Rosin will oxidize at ordinary temperature. Finely ground rosin will oxidize and contains more water after exposure in an open room.

Mr. Romaine. Mr. E. V. Romaine.—Some time ago I had occasion to perform an experiment of that kind. The amount of water I got after exposing it for several months was very minute. Ordinary measurement did not give it. The rosin was in powder form. I believe the article by the Goodyear Rubber Co. shows that oxidation takes place at 180° C. It is possible that the oxalate which is formed may be the cause of this trouble, because at 180° C. in a vacuum or in ordinary atmospheric pressure, I believe the water formed would be evaporated.

Mr. Walker. Mr. Walker.—If oxidation does not take place at ordinary temperature, why is it that pure abietic acid will be very dark colored on a very short exposure to the air?

Mr. Romaine. Mr. Romaine.—A very small percentage of some unidentified impurities will cause that reaction, more than the actual oxidation.

Mr. Walker. Mr. Walker.—But it does not take place in the absence of air. If pure abietic acid is sealed in hydrogen, it will not turn brown.

Mr. Romaine.

Mr. Romaine.—That is perhaps similar to naphthalene, alpha-naphthylanim or other organic compounds. The presence of an almost undeterminable amount of impurities will cause oxidation. Without the presence of those minute quantities of impurities, it will not happen. I have had some pure abietic acid exposed to the air for some time without oxidation.

Mr. Johnsen.

Mr. A. M. Johnsen.[1]—I should like to ask whether the presence of oxidized rosin in the rosin as it is sold to the electrical insulation manufacturers is considered objectionable or not. If the rosin is already partially oxidized, do you consider it objectionable?

Mr. Hanson.

Mr. Hanson.—I can not answer that.

Mr. Johnsen.

Mr. Johnsen.—My point is that abietic acid is an unsaturated compound and, as we understand it, it is possible to oxidize through different stages—you can have different degrees of oxidation. What degree of oxidation would you consider the critical point, if there is any?

Mr. Hanson.

Mr. Hanson.—I can not answer that question because I have no way of knowing exactly how to state this degree of oxidation. The way oxidation manifests itself to me is in the falling off of the electrical resistivity. I might say this, that for average use, if the compound starts out with a resistivity of 3×10^{12} ohm-centimeter, we can use it until it gets down to about 0.7×10^{12} ohm-centimeter at 85° C., and still meet the demand of the trade for average voltage cable, but not for 132,000 volt cable.

Mr. Logan.

Mr. W. B. Logan.[2]—Our experience with water in rosin runs something like this: It may be found in any one of three forms. One form is that water which clings to the rosin when it is rained upon, and it can and does get into the melting-down kettles and causes foam. Another form is that which is occluded by intimate moisture with the rosin, and causes the rosin to become opaque. This will cause foam, but it requires a very small amount of moisture to make rosin opaque —as little as 0.1 per cent will do so. Generally, opaque rosin does not foam excessively as moisture in melting down is released more gradually. One of the prime causes of opaque rosin is the mositure that the rosin takes up from the wooden package, and the use of the metal container generally eliminates this; also the metal package prevents rain from coming in contact with the rosin, and we have therefore advocated the use of metal packages.

There is a third form of water in rosin that causes the electric resistance of the rosin to be less than what it would be if the water

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[1] Engineer of Tests and Chemist, The Pullman Co., Pullman, Chicago, Ill.

[2] General Manager, Acme Products Co., Inc., DeQuincy, La.

Mr. Logan. were not present. We can not say definitely just how this water is bound up with the rosin, but it would resemble a slightly hydrated combination. We know that amorphous or clear rosin that has no occluded water such as would cause opacity if heated to approximately 175° C. will give off small quantities of water for a while, the amounts being from 0.2 to 0.4 per cent of the weight of the rosin, and that when it has ceased to give off more water, the electric resistance of the same rosin has been increased. Therefore, the electrical leakage is lessened.

Mr. Romaine. MR. ROMAINE.—In part of this discussion it was mentioned, I believe, that in one compound rosin was mixed with paraffin to form a wax. May I ask if at any time difficulty was experienced in obtaining a homogeneous mixture of rosin and wax? If so, what grade of rosin gave the most satisfactory results and what properties were the cause of the difficulty?

Mr. Roman. MR. ROMAN.—We have used it in one given proportion and have had no trouble with it.

ROSIN FOR THE FLOOR COVERING INDUSTRY

By R. B. Rohrer[1]

Although it was suggested this paper include a résumé of the use of rosin in the roofing and oilcloth industries, it will be noted that they are not included in the title. This is because it was found that little, if any, rosin is used in the former, and that used in the latter will be fully covered in the paper on varnish. A well-known authority, in a private communication states that "the roofing industry does not customarily use rosin in its manufacturing processes" and that "it might be well to clearly point out this fact." The rosin that goes into oilcloth is used almost exclusively as a varnish, either with or without pigment, a discussion of which would involve unnecessary repetition. This paper is accordingly confined to the floor covering industry only.

The Grades Used and the Reasons for the Choice of Such Grades

It is practically certain that wood rosin is almost universally used for the linoleum cement and for the cheap backing and coating paints. These include over 90 per cent of the possible outlets for rosin in this industry. Some gum rosin is occasionally used, but apparently not with great satisfaction, as the following quotation from a recent letter well illustrates:

> "We have used 'E' grade gum rosin. . . . Our experience has been that the grading is, in general, very poor, and occasionally shipments are received which contain a very large quantity of sticks, stones, and other dirt."

Little can be added to this except that repeated experiments have not shown any advantages of gum rosin, regardless of the grade, over wood rosin for the production of linoleum cement.

The reasons for this extensive use of wood rosin are very ably summed up by another engaged in this industry, in his reply to the author's request for opinions to be incorporated in this paper. His reasons are as follows:

> "Preference for this grade is due to the fact that we find this rosin to run quite uniform as regards melting point, acid number and color, and also find it uniformly clean and free from foreign materials. Naturally, the price consideration has considerable influence, particularly since the dark color of the rosin is not objectionable."

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[1] Chemical Engineer, Armstrong Cork Co., Lancaster, Pa.

EFFECT OF DIRT

Ordinarily the dirt occurring in wood rosin is not extremely detrimental, and complaints due to too much dirt are probably very few. Since the rosin is melted in very large quantities and generally strained through a screen in the melting kettle, the main difficulty from foreign materials is the clogging of the screens and the possible insulating effect of large accumulations on the bottoms of the kettles.

If, however, the amount of dirt is excessive or very dark in color, it may discolor the finished product or, as has already occurred, actually cause serious loss due to dark streaks on a product which should have a plain light-colored surface. This is very objectionable because considerable quantities of the cement will have been made before the difficulty is discovered, since it is customary to age the cement as much as several months before it is used. Formerly the charred chips of wood, sometimes found in the lower grades of gum rosin, were the chief offenders in this respect. Occasionally long, thin strips or splinters of wood will slip through the screens and cause damage, but it has always been difficult to establish whether they came from the rosin itself or from the barrels having been carelessly opened.

MELTING POINT

Rosin for linoleum cement is melted either in the cement kettle or in separate, continuously heated supply kettles. In both cases the heating is done by means of steam, either in a jacket or by means of coils. It will therefore be seen that unusually high melting point or slowly fusing rosin might readily slow up production, and there have been cases where partial solidification of the melted rosin has occurred upon the addition of the cold solid oil. Of course, the latter difficulty is no doubt more frequently caused by using rosin not sufficiently hot than by its being of unusually high melting point.

On the other hand, it is conceivable that too soft a binder would result from a rosin of too low a melting point. This possibility is specifically mentioned by another correspondent who further adds that "the higher melting rosin is preferred to the softer for several reasons, among which may be mentioned less loss due to running from the barrels in hot weather and less volatile matter lost in melting and in cement cooking."

In the manufacture of the backings and coatings, the melting point probably has little effect. It is claimed by some that the differences in melting points have an effect on the hardness and brittleness of the dried films, but the author has been unable to confirm this, at least in so far as normal variations in similar grades of rosin are concerned.

PHYSICAL AND CHEMICAL CONSTANTS

Owing to the complex nature of both rosin and linseed oil, particularly after the latter has been highly oxidized, the exact effects of the various physical and chemical constants are not known. The value of rosin in forming the linoleum cement is dependent on its property of peptizing, dispersing or "dissolving" a dried film of linseed oil. This "solution" is effected in steam-heated kettles and forms a sticky, cohesive, workable mass out of the otherwise worthless dried linoxyn. At the time of solution it also permits the addition of other materials such as the harder gums. Further, it continues, throughout the life of the finished product, to act as a sort of extender for the oil, thus preventing excessive hardening and helping to maintain the extraordinary flexibility of linoleum indefinitely, which is one of its chief characteristics. As an illustration of this effect, samples of linoleum in actual service over forty years were found to be quite as flexible as goods recently produced.

This property is no doubt due to the acidity of the rosin, but just why other organic acids, or materials of an acid nature, are not as efficient, is not known. Also, it has been found that the differences in acidity ordinarily occurring in rosin are without effect on its "solvent" powers, and, while these powers can be reduced by altering its acidity, the amount of alteration necessary is entirely out of proportion to its effect.

In the case of the coating paints, where lime hardening is the usual practice, the acidity is of undoubted importance, but for linoleum cement it is the general consensus of opinion that at present there are no chemical constants which measure its value.

As pointed out above, the physical properties, such as melting point and color, have certainly a bearing on its applicability, but tests for these qualities in rosin could easily be over-emphasized. The optical rotatory properties have not been investigated by the writer and apparently not by any other linoleum manufacturer.

CRYSTALLIN ROSIN

In the use of wood rosin there is always found some small quantities of the crystallin variety, but, out of a probable consumption of some twenty million pounds during his experience, the author has observed less than a total of 10,000 lb. of accumulation of unusable material which could be classed as crystallin—about 0.05 per cent. This is a very small percentage and it occurred in such a way that no difficulty was experienced with it and was only found on cleaning kettles which had been continuously used for a considerable length of time. It is possible that a considerable proportion of this was actually formed in the kettle from rosin which was amorphous when received.

Crystallin rosin is supposed to have a higher melting point than the amorphous variety and, presumably, to be less soluble in oils and thinners. We have observed very little that was infusible when aided by the fluxing action of the usual preponderance of amorphous rosin. Furthermore, we have purposely tried a barrel of very crystallin rosin, submitted by a manufacturer, with no notable difficulties. The product was equal to, and could not be distinguished from, a companion piece made from the regular run of rosin.

But it must not be supposed that large quantities of crystallin rosin could be used with impunity. We had occasion to submit a sample of an accumulation of material from one of our kettles to the producers which they reported as having "every appearance of being crystallized." Although this concern determined its melting point as 186° F. (75° C.), it remained unmelted in a kettle of molten rosin heated with ninety pounds of steam. Its specific rotation was reported as +3.7.

On the other hand, we cannot help but feel that it would be inadvisable to make crystallin wood rosin a separate grade on account of the rare occurrence of excessive quantities and the fact that, in our own long experience, we have never made a single complaint for this reason except for the water which so frequently causes, is caused by, or merely accompanies this variety of rosin. This water, it should be added, is often the source of considerable difficulty in our melting kettles as it causes foaming which is difficult to control.

INTERCHANGEABLE SUBSTANCES

Materials that are or might be interchangeable with rosin should be put in two classes: (1) Materials to be substituted for rosin, and (2) materials for which rosin may be substituted.

1. As no other gum or resin so far produced compares with rosin in price, there has been very little competition with it for use in linoleum, while also, so far as we know, no other resin would be quite so satisfactory for reasons outlined above.

Many other resins have been tried, and even patented, but none is apparently in use at present in the manufacture of linoleum. Only the coumarone resins seem to offer any prospect of competing, in the near future, in the matter of price. Their alkali resistance is an extremely important asset which is only counterbalanced by their inferior "solvent" action on linoxyn. The writer has made and seen made a number of apparently very good pieces of linoleum with various quantities of these resins. The bakelite, albertol, and other condensation type resins have found application only in certain linoleum substitutes made in Europe, and, although many patents have been taken out covering their use, none is at present employed. Their price, like those of the many natural resins, preclude their extended use.

2. On account of its softening effect on linoxyn there is a limit to the proportion of rosin that can be used. In order to overcome this defect it has been the custom, since the very beginning of the industry, to use with it Kauri gum. Many substitutes have been tried for this material but none has been entirely satisfactory. The available supplies of this valuable gum are reported as being greatly depleted, and since the war prices have steadily gone up. It would appear that extensive research on the part of the rosin producers might yield a profitable substitute for this valuable, but apparently vanishing, raw material. When it is considered that the price of even the crudest usable grades of this gum is four and five times the present price of rosin, it would seem that the latter could receive quite an elaborate treatment and still easily compete provided its properties approached those of the fossil gum.

CONCLUSIONS

It is obvious from what has been said, therefore, that the prospects are good for the continuance of the favorable position that rosin, especially wood rosin, now enjoys in the linoleum industry, and that there are some excellent opportunities for increasing the proportion now used in the production of floor coverings. It is equally obvious, however, that synthetic resins have recently made some very threatening gestures against this position which, though strong, is by no means impregnable.

To summarize, in conclusion, the properties essential to rosin for the floor covering industries, the following list is given in the order of decreasing importance:

1. Ability to "dissolve" linoxyn;
2. Absence of water;
3. Light color consistent with price;
4. Freedom from dirt, especially sticks and splinters of wood;
5. Uniformity of melting point;
6. Absence of the crystallin variety;
7. Acid number.

From an economic point of view the question of price is, of course, an essential consideration, and the author would rank it second in importance only to the "ability to dissolve linoxyn" in the above list of essential properties.

Further, to make rosin more acceptable to this industry, all the above essential properties could be improved, and increased usage could be obtained by giving it properties similar to those of Kauri gum.

Specifications for use in buying and selling, particularly the present excessive number of grades, should be simplified as much as possible and no difficult tests, or any of doubtful value, should be introduced.

The Committee on Naval Stores, therefore, should be extremely careful that no complicated or unnecessary specifications are adopted and one of its chief concerns should be to reduce the present number of classifications into which rosin is divided.

Acknowledgement.—The author is greatly indebted to Messrs. Herbert Abraham of the Ruberoid Co., John E. Pyron of the Certain-teed Products Corporation, R. D. Bonney of the Congoleum-Nairn Co., and H. G. Hassenplug, of the Geo. W. Blabon Co., for their co-operative suggestions and the information furnished by them which has been incorporated in this paper.

DISCUSSION

Mr. Logan.

Mr. W. B. Logan.—Mr. Rohrer mentioned the acid number as one of the criterions. Can he say in what way the acid number is of importance?

Mr. Rohrer.

Mr. R. B. Rohrer.—Apparently the property of rosin which causes its peptizing effect on the oil film is in some way related to its acid number, although, as stated in the paper, we could not trace this dispersing effect in any one grade of rosin directly to its acid number, at least in so far as normal variations in this constant are concerned. If rosin is neutralized with lime, zinc or glycerin its dispersing powers will be impaired, but not to the extent one would expect if the acidity were the whole cause. All we can say at present is that there is a general relation between the acid number and its usefulness for our purposes.

Mr. Logan.

Mr. Logan.—You mean an acid number of 170 would be more satisfactory than one of 150?

Mr. Rohrer.

Mr. Rohrer.—It is difficult to say definitely. Theoretically the 170 acid number should be more suitable but full proof is lacking. It is probable that if the acid number were reduced to as low as 100, by special means, the difference would be marked.

Mr. Logan.

Mr. Logan.—May I ask what effect coumarone has?

Mr. Rohrer.

Mr. Rohrer.—As mentioned in the paper, we cannot use one hundred per cent coumarone, but it can be substituted for a portion of the rosin.

Mr. Logan.

Mr. Logan.—In substituting it, does it affect conditions in anywise proportionately with the amount used, or in other words, in relation to the lower acid number?

Mr. Rohrer.

Mr. Rohrer.—The decrease in acid number due to substitution of a non-acid substance is probably the cause of a decrease in some qualities and is probably the reason we could not use all coumarone.

I should like to repeat that coumarone has the advantage of being alkali proof. That is quite an advantage because, do what we will, we cannot always prevent the housewife from using highly alkaline cleaners although she would not think of using them on varnished or painted surfaces.

Mr. Logan.

Mr. Logan.—Would not a rosin of low acid number then be of similar benefit to you in floor coverings, just as an ester is of value in the spar varnishes due to its property of not blooming or whitening up?

Mr. Rohrer. MR. ROHRER.—It depends to some extent upon what is the cause of the lower acid number. If it is due to unsaponifiable matter there may be some increased resistance to water and alkali. If it is due to having esterified the rosin with metals or glycerin, little advantage would be obtained. I believe, however, that the protective influence of coumarone is greater than most unsaponifiable materials that might be added. A distinction can be made between protecting saponifiable materials and merely diluting them with inactive material.

Mr. Logan. MR. LOGAN.—Your opinion then is that if an ester gum were used instead of rosin in an uncombined state, sodium carbonate would affect it?

Mr. Rohrer. MR. ROHRER.—It would be quite as bad as the acid rosin.

Mr. Breyer. MR. F. G. BREYER.[1]—I know little about rosin but on this question of acid number, we have made quite a few experiments and it seems that so far as the ability to cut consistency is concerned, there is a general relationship between the acid number of any organic material and its ability to cut consistency. It does not make any difference whether it is lynolic acid, abietic acid or what, there is a definite relationship. In our experience the strongest consistency cutting acid is sulfur dioxide.

There are two things about acids and their ability to cut consistency; one is the strength of the acid and the other, its solubility in the material into which it is to be put. I should hesitate to believe that in rosin the cutting power was not proportional to the acid number. If high and low acid number rosins were found having the same cutting power, I should insist that it must first be shown that something else has not entered into the rosin to neutralize the higher cutting value of the higher acid number.

In addition to acid number and solubility, there is the question of composition. An organic acid has in general much more cutting power on organic compounds than most inorganic acids, but there are exceptions to that rule, too.

For example, take rubber. Rubber can be thinned by adding some mineral oil to it. That will thin the material down, but it will still be what the paint man calls "puffy." But when rosin is put in it, it loses its yield value. One is pure dilution with a medium of lower consistency, the other is a cutting of the consistency. That is associated with acid number.

I should go one step further and say it is the conclusion of the workers in our laboratory that the maximum consistency of most organic two or more phase systems in the nature of rubber or plastics

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[1] Chief, Research Division, New Jersey Zinc Co., Palmerton, Pa.

Mr. Breyer. is attained at neutrality, that in practically every case acid results in lowering the rigidity of the material (rigidity being proportional to the force required to produce flow).

Mr. Rohrer. MR. ROHRER.—There are several reasons why we have been unable to pin down this dispersing power of rosin directly to acid number. One is, there is no doubt but that we use, in the majority of cases, rosin in excess of the amount required to actually peptize the dried film. Secondly, there are so many side reactions, including combinations between oil, rosin and gums, which obscure attempts to correlate the two properties. In this discussion there has not been a clear distinction between acid number which has been purposely changed and the small differences normally occurring in different runs or shipments of the same grade of rosin. It should be clearly understood that great differences in the quality of linoleum can be effected by purposely changing the acid number, as well as other characteristics, of resin content, but that the differences caused by the comparatively small variations of well made rosin are not sufficient to be detected by ordinary means although it is probable that some differences do and theoretically should exist.

ROSIN FOR SOAP MAKING

BY F. J. ARTHURS[1]

In soap manufacturing, rosin is used principally in yellow laundry soaps, that is, its use has not been common in other soaps or soap chips. The rosin should be clear and true to color throughout each package. Moisture is objectionable. Dirt is not acceptable. Opaque rosin is not desirable on account of the uncertainty of remelted color. In other words, soap manufacturers are vitally interested in what quantity of dry, clean, clear rosin they have to use in soap for a given expenditure.

Generally speaking, rosin has been used in soap manufacturing as extensively as the grade of soap would permit, just as long as it cost less per net pound at the soap kettle than any fat that could be secured to incorporate into that same grade of colored soap. Advances in cost per net pound of rosin in recent years have had a great deal to do with the introduction of white or light-colored laundry soaps and chips. Many fats at present cost less per net pound at the soap kettle than a net pound of rosin of "H" or lighter grade.

Soap manufacturers realize the difficulties encountered when trying to regulate packages, labor, equipment and the quality of such a crudely produced product as rosin. Certain skepticism has always existed in relation to rosin in the soap trade, but better understandings between producers, factors, marketers and consumers are encouraged and are increasing.

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[1] Larkin Co., Inc., Buffalo, N. Y.

DISCUSSION

Mr. V. E. Grotlisch.—I should like to know how the dirt in rosin is gotten rid of in making soap. **Mr. Grotlisch.**

Mr. F. J. Arthurs.—Soap is thoroughly boiled, of course. The rosin may go right out of the barrel into the soap kettle, broken by an axe or other method, or it may go through a screen into a tank where it is saponified into a liquid. In that boiling process the chemists all know there is a settling out or salting out which takes out everything except these lighter-than-soap chips, which float and are the ones which you sometimes get in the cake. **Mr. Arthurs.**

Mr. Grotlisch.—Then rosin that contains sand would not be nearly so objectionable to you as a rosin that contains bark and wood which would float? **Mr. Grotlisch.**

Mr. Arthurs.—We do not like to have sand delivered on rosin purchases. Excessive water, sand, dirt, etc., is not acceptable as rosin and we reject such deliveries when we buy rosin. **Mr. Arthurs.**

Mr. Grotlisch.—My idea was not to sell sand for rosin, but we of the Bureau of Chemistry are working on this matter of determining dirt in rosin and how much is objectionable for certain purposes, and we should like some information on the subject. **Mr. Grotlisch.**

Mr. Arthurs.—We object to all dirt, but we tolerate what is practical and can not reasonably be avoided in the manufacture of any crude product such as we consider rosin to be. **Mr. Arthurs.**

THE USE OF ROSIN IN PRINTING INKS

By Louis M. Larsen[1]

Rosin is used quite extensively in the printing ink industry. It is a solid material of high acid content, and therein lie some of its uses and some of its objections.

Rosin, when incorporated in varnishes and oils under the influence of heat, produces an increase of viscosity. "F" rosin shows slightly greater bodying power than does "WW" rosin, both of which are used. As a thickener, it is used mainly because it is cheap, and any cheaper gum of similar thickening properties would, of course, displace rosin in this field. Cumar gum of high melting point is now more expensive than rosin, but gives a greater body than rosin. In order to determine the bodying effect of rosin, one part of the sample is dissolved in two parts of thin litho linseed varnish at or about 130° C. After cooling, the viscosity may be determined at room temperature by any standard method. A very rapid method of showing the general character of rosin is to allow the standard solution and the sample solution to penetrate machine finish paper, beginning at the same instant of time. The penetration through the paper is observed on the opposite side, and a qualitative measure of the body may be obtained in that manner.

Due to the high acidity and to the specific character of the rosin acids themselves, reactions with basic pigments, such as white lead, occur, resulting in the formation of rosin soaps. Continued action, influenced greatly by temperature and time, results finally in a decided increase in viscosity, transforming the original soft ink into a hard useless solid. As the rosin acids readily combine with many of the commercial pigments, rosin is objectionable in such inks. Rosin transformed into ester gum possessing low acidity, shows far less chemical action with basic pigments, and it is likely that this process will be required to render rosin safe from heavying up the ink. Likewise cumar gum, showing low or no acidity, parallels ester gum in action upon basic pigments and can be used in inks.

While both rosin and cumar gum possess bodying power, only rosin has drying power—a valuable property indeed. Combined with metallic driers, either in conjunction with linseed or other drying varnishes, or even in conjunction with non-drying oils (such as paraffin oil), it oxidizes quite rapidly to a hard film when the ink is printed on paper. In order to determine the drying power of rosin one may proceed as follows: Dissolve one part by weight of rosin in three parts of 18° Baumé mineral oil, and grind in this varnish one part of carbon black. After adding a predetermined quantity of drier, the ink is proved on paper for drying test observations. After three or four hours the proofs are examined by a finger rubbing test every half hour until results show whether or not the sample is up to the standard. Ester gum dries even faster than rosin itself, and both of them faster than rosin oil.

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[1] Chemist, The Ault and Wiborg Co., Cincinnati, Ohio.

Of course, rosin being a solid is useless in this form as a vehicle directly for printing inks, but either must be dissolved in some solvent, such as linseed varnish, or be converted into a new product, called rosin oil, by a destructive distillation process. Rosin is thus converted to rosin oil, a viscous material which, when properly handled, retains considerable of the original drying property, that is, drying by oxidation. Rosin oil can be procured in varying viscosities, and when combined with the original solid rosin by heat treatment it is possible to obtain varying degrees of drying and body. Rosin oil darkens somewhat with age and therefore is not suitable for whites, yellows, reds and other bright pigments. Besides, it shows the same type of soap formation with basic pigments as rosin itself does, and inks often heavy up. Thus the drying power of rosin can be utilized directly in combination with other oils, or can be utilized indirectly as rosin oil prepared from rosin.

In the manufacture of driers, the metallic catalysts (cobalt, manganese or lead) may be combined with rosin to produce oil-soluble soaps possessing high catalytic power. These are manufactured from rosin, either by a water-precipitation process, or by a varnish-heat process. The former employs rosin, caustic soda and a water-soluble metallic salt; the latter employs rosin and a corresponding metallic oxide or hydroxide. Because rosin is relatively cheap, driers are frequently made from it, producing a high quality drier at a low cost. Rosinates as a rule are quite solid, while corresponding linoleates are semi-viscous and difficult to handle.

The presence of dirt influences the color of varnishes, especially when heated to a high temperature. Only clean pieces of "WW" rosin are selected for very pale varnishes. Settling of the finished varnish, or centrifuging, while warm removes the dirt present.

We have had very little experience with wood rosin, and therefore cannot make a report at this time.

As the most important properties of rosin for printing inks are drying and bodying power, the usual differences in melting point, content of dirt, and slight difference in color are generally overcome by slight adjustments in the varnish formula and in handling. Rosin shipments, with few exceptions, have been found up to standard in both drying and bodying power.

DISCUSSION

Mr. V. E. Grotlisch.—It is interesting to note that Mr. Larsen says that "F" rosin, which has a slightly lower acid value than Water White rosin, has greater bodying power. In other words the material with lower acid value does not obey the general law that the greater acidity goes hand in hand with greater consistency-cutting power. **Mr. Grotlisch.**

Mr. F. G. Breyer.—If rosin is added to a homogeneous single-phase fluid, such as kerosine, the consistency of the kerosine is increased simply by diluting with a thicker medium. If rosin is added to raw linseed oil, which is very close to being a homogeneous single-phase system, its viscosity is increased. However, if linseed oil is taken and a two-phase system made of it by bodying it up in any manner whatever or by making a secondary phase, by blowing it or putting a finely divided material in it like lampblack or zinc oxide, it will be a two-phase system which has a given solidity factor. If rosin is added, it is the wetting or dispersing power of the rosin which disperses or deflocculates the dispersed phase and cuts the consistency. That applies to all materials that we have been able to examine microscopically that are two or more phase systems. **Mr. Breyer.**

In other words, we do not cut the body of a single-phase system by adding rosin. We either increase the consistency if it is lower than rosin, or decrease the consistency if it is higher than rosin, but if it is a two-phase system, we will cut the consistency.

THE USE OF ROSIN IN THE MANUFACTURE OF CORE OILS

By W. R. Pate[1]

Core oil that is clear and light in color is far more salable than oil dark in color, although the binding strength of the oils may be the same. This fact has worked to the advantage of gum rosin for years, as the manufacturer could obtain real light-colored core oils by using gum rosin, although there was no gain in tensile strength. Recently, the wood rosin industry has made remarkable strides in producing rosins of lighter color and I understand that it is now possible to make such wood rosins. The author has noticed a tendency on the part of wood rosin to darken in color on prolonged heating much quicker than gum rosin does.

Some manufacturers claim that gum rosin gives better binding strength than wood rosin, but the author has made several hundred batches of core oils out of both grades, made test bars from these batches, and on obtaining the breaks and averaging the results did not find any appreciable difference.

There are other reasons besides color that cause a large number of manufacturers to prefer gum rosin. One in particular is that rarely is any trouble experienced from crystallization, whereas wood rosin has often crystallized nearly entirely out of the core oil and caused considerable loss. In spite of the fact that crystallization is not fully understood, the wood rosin industry has the difficulty well in hand at the present time, but the old adage "a burned child dreads the fire" still applies. Secondly, a flocculent precipitate is occasionally obtained from wood rosin which has the appearance of aluminum hydroxide and is commonly referred to as "nigger." This precipitate does not form until the oil is filtered and allowed to cool thoroughly. This difficulty has never been experienced by the author with gum rosin.

There has been a great deal of investigation by the large consumers of core oil and a few of these have drawn up their own specifications. They invariably insist upon very high acidity, saponification and iodine values. The manufacturer that can make an oil having these values at the cheapest price secures the business, and as rosin, in normal times, is much cheaper than drying oils, the rosin with the highest acidity, saponification and iodine values is the most economical to use. On these tests, wood rosin averages much lower than gum rosin. There are such tests as color, viscosity and gravity that prevent the manufacturer from increasing the percentage of rosin.

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[1] Lindsay-McMillan Co., Milwaukee, Wis.

All of the advantages are not on the side of gum rosin, since the production of wood rosin is centralized, and under strict laboratory control a far more uniform and cleaner product is obtained. For instance, a batch of core oil made from gum rosin filters much more slowly than a batch made from wood rosin due to the presence of a great deal of clay in suspension, and sometimes it is almost impossible to catch this clay on the filter. In addition to this, it is necessary to clean out the kettle after each batch of core oil on account of clay, pebbles, sticks, etc., which settle to the bottom. At one time last summer the author found 50 lb. of such material in one barrel of gum rosin.

There is a definite relation between the melting point of rosin and the viscosity of core oil, and for the manufacturer who wants his physical factors to be a constant, wood rosin has the advantage.

An important point for core-oil manufacturers is the tendency of some core oils to cause sticking in the core box. This is a serious handicap. The writer has not been able to obtain as accurate data on this as he would like. A rosin of low melting point increases the sticking, as a general rule. There is not the tendency in wood rosin to cause sticking as much as in gum rosin; the darker the grade of gum rosin the more pronounced is the sticking.

There are many other factors that could be brought up, but the author feels that the main points have been covered and offer opportunities for further discussion.

DISCUSSION

Mr. Grotlisch. MR. V. E. GROTLISCH.—In the last paragraph of Mr. Pate's paper is the statement: "A rosin of low melting point increases the sticking." I wonder if that is due to the unsaponifiable matter. We know there is somewhat more unsaponifiable matter in the darker and lower grades than in the higher grades.

Mr. Pate. MR. W. R. PATE.—I am not able to answer that. In fact, the longer I work with rosin the more fertile the field for research in it appears to be. In the core oil industries there are quite a number of interesting problems to be worked out, and that is one of them.

Mr. Grotlisch. MR. GROTLISCH.—We know the acid number of the lower grade rosin is somewhat lower than the acid number of the high grade and also the unsaponifiable matter in the low grade rosin is higher. That unsaponifiable part of rosin, which is called resene, is the sticky part of rosin.

Mr. Pate. MR. PATE.—I should like some discussion on the amount of water. I think that in working with gum and with wood rosin you will find more tendency of gum rosin to split off water than wood rosin. That has been my experience. We have had cases of heating up wood and gum rosins to say 300° F. and then letting them cool and finding various percentages of water when the material had cooled. I think gum rosin gives out water more readily than wood rosin. At least water is formed; it may be by oxidation.

THE KIND OF ROSIN THE ROSIN OIL MANUFACTURER WANTS

By Brian S. Brown[1]

To the manufacturer of rosin oils the most important specification for his rosin can be expressed in one word—cleanliness. Peculiarly, the low grades, B and D, produce the richest oils and these are the very grades the producers are prone to be most careless in straining. The facts that these are the cheapest grades, that the inspector cannot degrade B, and D but one grade, and that the darkness of color of these rosins makes it difficult for the inspector to see dirt, makes the producer careless of cleanliness to a point that the rosin oil manufacturers suffer.

Rosin oil is made by the destructive distillation of rosin in cast-iron stills, a residue of black pitch in amount of approximately 3 per cent remaining in the still. After each charge this is drawn off, but regularly after a periodical number of runs, it is necessary to close down the stills, let them cool, and then with chisels clean out the conglomeration of dirt and pitch, nearer to concrete in its properties than to anything else. The number of charges which can be run before closing down is inversely proportional to the amount of foreign matter in the rosin. To one familiar with the loss of efficiency from such a shutting down no more need be said.

As stated above, the richest oils are produced from the darker grades of rosin, but the rosin must not be burned in the process of manufacture. This produces a rosin which will make a rosin oil of thin body and easily oxidizable properties, which would be very objectionable when used in a light-colored axle grease. The test for the rosin in this case is to take a piece about the size of a pea and crush it to a powder. Genuine gum rosin, no matter what the grade, will give a white powder. If there is a tendency to fail to grind to a dry powder, or the powder is yellow or brown, let the rosin oil manufacturer beware. The acid number of a good grade of gum rosin should run around 162. This for the benefit of the man who wants to give a technical turn to his specifications.

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[1] President, Georgia Rosin Products Co., Brunswick, Ga.

DISCUSSION

Mr. Pate. MR. W. R. PATE.—We used to buy rosin oil to make axle grease but the last four or five purchases of rosin oil would not work. The oil would set and then suddenly turn mushy again. Is there not adulteration now in the rosin oils?

Mr. Brown. MR. B. S. BROWN.—There is and always has been. You can buy rosin oil that has been adulterated that may or may not set. The largest manufacturers of axle grease in the United States use an oil that has a certain amount of petroleum oil in it. They buy such an article knowing of the adulteration. They can make a test for it. By a certain process it is used and fluxed and it makes good grease.

Mr. Pate. MR. PATE.—I know quite a number of grease makers are getting a little skeptical about the rosin oil they buy. They seem to be having trouble lately, in the last six months or a year, because the rosin becomes mushy. Now they can buy some fatty acids double distilled from garbage that are not so very different in price from rosin, and quite a number are getting away from using rosin oil because of the trouble experienced.

Mr. Brown. MR. BROWN.—If you are uncertain whether you get pure oil or not, I shall be glad to give you a test to show whether it contains petroleum oil or not.

Mr. Veitch. MR. F. P. VEITCH.—Is it not customary to make such a test? I think it is not difficult to determine the presence of petroleum oil.

Mr. Brown. MR. BROWN.—Any considerable amount of adulterant is easily found. Three or four or five per cent is hard to find, but there is a test that determines very easily whether petroleum oil is present.

Mr. Riemersma. MR. M. J. RIEMERSMA.[1]—Wood rosin in distillation gives lower acid number than gum rosin.

Mr. Brown. MR. BROWN.—I am aware of that fact and have not been able to use wood rosin in the manufacture of rosin oil for that reason. I have been distilling wood rosin off and on for about thirteen years and it is my invariable experience that it will give a lower acid oil, and while I am located right next to the largest wood rosin plant in the United States and should like to use it, my consumption is comparatively small.

Mr. Grotlisch. MR. V. E. GROTLISCH.—Will you tell what you mean by a "rich" oil? You say the low grades "B" and "D" produce a rich oil.

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[1] Hercules Powder Co., Rotterdam, Holland.

MR. BROWN.—By a rich oil we mean one with a higher acid number. **Mr. Brown.**

MR. C. E. KINNEY.—We should like to know why wood rosin cannot be used for rosin oil satisfactorily. The acid number of wood rosin is about ten lower than that of gum. That is, the acid number of gum rosin is about 162 and that of wood rosin is about 152 or somewhere around there, but the difference in acid numbers of the rosin oil is about twenty, or more perhaps. Does it seem reasonable that a difference of ten in the acid numbers of the rosins should give such a difference in the rosin oils? **Mr. Kinney.**

MR. BROWN.—I might say the unsaponifiable matter that we have to start off with, the resenes, probably come over. They have no acids in them at all. They come over *in toto*. If you should start off with the acid number 150 and cut it in two you have 75. You start off with 160 and cut it in two and you have 80; but if you have a constant there, ten or fifteen points, perhaps, you will get a larger percentage of unsaponifiable matter in the rosin oil from wood rosin. That is simply a theory with nothing to substantiate it. **Mr. Brown.**

MR. E. V. ROMAINE.—Do you not think possibly it is a physical rather than a chemical property of the rosin? I believe four or five years ago the Standard Oil Co. of Indiana patented a process whereby it made grease set oil from gum rosin which was not subjected to distillation but was heated at about 325° C. for, I believe, 22 to 24 hours. The acid value of this material was materially decreased. However, the reset property was increased. I believe the process is being used to-day. **Mr. Romaine.**

MR. BROWN.—I am not able to give an explanation. Was wood rosin tried out? **Mr. Brown.**

MR. ROMAINE.—This was all gum rosin. **Mr. Romaine.**

ROSIN FOR THE MANUFACTURE OF STICKY FLY PAPER

By B. E. Kuyers[1]

The author does not know of any use for rosin in the insecticide field except for the manufacture of sticky fly paper. Rosin for this purpose should be E or F grade, of fairly uniform melting point, clear, and free from dirt.

The greatest difficulty in getting rosin with these simple requirements is to secure it free from dirt, but this difficulty is met by straining the sticky material through cotton.

There is no practical substitute for rosin in making fly paper sticky.

I regret that I cannot go into greater detail, but our formulas and methods of manufacture have always been secret, which makes it very difficult to prepare a paper on the subject.

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[1] Chemist, The Tanglefoot Co., Grand Rapids, Mich.

HOW GUM ROSIN IS MADE

By C. F. Speh[1]

Rosins and turpentines are divided into two general classes: those obtained from the crude gum which has exuded from the living tree, and those obtained by various processes from the dead down, lightwood and stumps. The first class is known as gum products: gum turpentine and gum rosin. The second class is known as wood products: wood turpentine and wood rosin; and depending upon the process employed in their production, the turpentines are again subdivided into destructively distilled wood turpentine and steam-distilled wood turpentine. The former process produces no rosin.

The gum products only are treated in this paper. These are produced by about 1500 producers in a belt reaching from North Carolina through the South Atlantic and Gulf States to the eastern border of Texas. Their size of operations varies greatly. Of the total production of gum turpentine, 11 per cent of the operators produce annually 100 barrels each and under; 40 per cent produce 250 barrels each and under; 80 per cent produce 500 barrels each and under. Partly included in this are a large number of operators who make a few barrels of gum per year, not sufficient to justify having a still of their own, and therefore they sell the gum to some other operator. This shows large percentage production by small operators. From this it can be seen that we are not dealing with a manufacturing plant or an establishment that can exert chemical control over its products. Further, under present economic conditions, the average place has such a short life that the expenditure of large sums for expensive equipment is not justified.

The production of gum rosin and turpentine may be described briefly as follows: The longleaf yellow pine produces the crude gum from which the rosin and turpentine are obtained. Beginning in March and ending in October, at least once a week the tree is "chipped," which consists in cutting away the bark and the sapwood to a depth not to exceed $\frac{3}{4}$ in. and for a height of about $\frac{1}{2}$ in. As a result of this wounding of the tree, there immediately starts up an exudation of the oleoresin. The crude gum is solely the resin acids dissolved in the volatile oil, spirits of turpentine. The gum flows over the "face" of the tree into a cup, the collecting receptacle.

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[1] Secretary-Manager, Turpentine and Rosin Producers Assn., New Orleans, La.

These cups are emptied at regular periods and the gum hauled to the still. The still is an extremely crude affair consisting simply of a copper kettle of about 20 bbl. capacity heated by direct wood fire. Many of the producers have installed recording thermometers in the stills to have better control of the distillation. The turpentine is volatilized and driven off with the water occurring naturally in the gum and any that may have been added. It is condensed and separated from the water by gravity. The rosin remains in the kettle and in its molten condition is strained through wire and cotton batting. It is then ready for barrelling.

It is entirely possible under present conditions to produce gum rosin that is clean and properly strained. The color may be controlled and is dependent upon the class of equipment used and the age of the face over which the gum must flow to reach the cup. The melting point of the rosin may also be controlled somewhat, because the producer can govern the amount of turpentine that he leaves in the rosin. However, it must be borne in mind that the longer the melted rosin is subjected to heat to distill off the turpentine and the higher the temperature, the lower will be the grade of rosin produced, with a corresponding reduction in market value. Here, then, we have the extent of the producer's control over the character of the product; namely, in cleanliness, melting point and color.

There is at present a slowly increasing recognition of the use of steam stills and it is entirely possible that as a result of the use of this more technical process we may be able to exercise some control over the chemical constants. This, however, is something for the future.

It is appreciated that all consumers desire clean rosin and that as a general rule they desire rosin of high melting point. The color desired depends entirely upon the product to be made. As just pointed out, these factors can be controlled as we are continually trying to do. It is appreciated that the soap manufacturer and paper-size manufacturer desire high saponification number; also that the varnish manufacturer desires particularly a high melting point rosin and would prefer a low acid number, and that if he had a rosin with these characteristics he would have a product which would practically replace many of the fossil gums in that industry. Rosins for other uses, such as for printing inks, sealing wax, core oils, linoleum, etc., require no characteristics other than those mentioned for the uses cited.

Because of the continually increasing cost of production, both as concerns labor and timber, and a widely fluctuating market over which he has no control, the rosin producer has in the main lost money. It is therefore rather difficult, at least for the present, to bring about the addition of any equipment which might be used to control constants other than cleanliness, color, and melting point.

There has been recently a gradual recognition of one objection to rosins and that is the tendency on the part of some rosins to crystallize. Apparently this tendency is becoming more prevalent, and it is probably due to the fact that the improvement in the distillation equipment permits the heating of the gum to a higher temperature, still obtaining the paler rosins. Work that has been done to date would indicate that the rosin acids are of three forms and may be divided into three stages as regards temperature and length of time of heating. The lowest temperature and shortest time of distillation apparently give rosin acids of the first stage, which have a slight tendency to crystallize. If the rosin is heated to a higher temperature or for a longer time at the upper temperature limit of the first stage, it seems to be converted to a form which does not crystallize. If, however, the rosin is heated at a still higher temperature or for a still longer time, it seems to be converted to a third form which crystallizes very easily. Additional research work is being done on this subject and it is hoped that as soon as the cause and limits are definitely determined, specifications for distillation can be set up and this tendency of crystallizing practically overcome.

The naval stores producers are rapidly realizing the need on their part for studying the requirements of the users of their products and they are generally trying to improve the quality of such products as far as any improvement is needed and can be made.

DISCUSSION

A Member. A Member.—What is the objection to putting up all rosin in metal containers?

Mr. Speh. Mr. C. F. Speh.—One objection is there are 1500 producers. About 11 per cent of them each produce 100 barrels or less of turpentine and about 350 barrels of rosin. To economically use a metal barrel it must go to the producer in a knock-down form, unless the freight rate should be such that an assembled barrel would not cost him too much. For him to assemble the barrel at his plant he must have some kind of crimping machine to crimp the sides and heads. That will cost him quite a bit. He may be established at one place for two or three years. The next year he may move five miles off and will then have to move the entire outfit. In the case of the small operator it is impracticable. On the other hand the big producer is in a position to handle it in that way.

I am not saying it cannot be done. If you should assume the attitude, which I hope you will not, that nothing but metal containers will do, naturally they will come.

On the other hand, one of the biggest objections to the metal barrel comes from the consumer. I think there is a summary in one of the papers indicating that nine consumers preferred metal and one preferred wood; one had no preference. I dare say if a canvass of all consumers were made, those who prefer wood would outnumber those who prefer metal. There it is a matter of education. For the average producer to educate the consumer is impracticable. Bear in mind also that the wood barrel is made from waste material at the saw mill. The mill owner is not going to encourage anything which will eliminate a market for waste material.

As it is now, both gum and wood rosin can be procured in metal barrels and those people who have had the patience to test it out find it is proving satisfactory. The metal barrels used are black metal, in most cases, and in some cases are lined on the inside. In other cases there is no preparation on the inside, but those have proved unsatisfactory. We are making some now out of galvanized sheet iron and it is possible they will answer the problem.

A Member. A Member.—Have you eliminated the possibility of water getting in?

Mr. Speh.

MR. SPEH.—Bearing in mind that these wooden barrels are stored in all sorts of weather you will appreciate that water cannot be entirely eliminated. Not only does water get on the inside of the barrel but any sudden change of temperature will crack the rosin, and if a rain comes the water will seep into the cracks. Then, if a hot day comes the heat will seal it in. I have seen cases where the barrel will be dry both outside and inside, but when the rosin is broken up you will find water. A crevice has been formed and there is water in it. It does not amount to much, but put into a hot kettle it will give some trouble.

WOOD ROSIN: ITS PRODUCTION, PROPERTIES AND USES

By J. E. Lockwood[1]

Production Process

Wood rosin is obtained from the pine stumps and waste wood of the cut-over lands of the South, by what is known as the steam and solvent process. This process consists essentially in reducing the wood to suitably sized chips, steaming the chips in digesters to distill off the turpentine and most of the pine oil, next treating the chips with a petroleum solvent to extract the rosin and the remainder of the pine oil, then evaporating the resulting crude solution to remove the solvent and pine oil, and finally subjecting the crude rosin to a special finishing treatment to more completely eliminate the residual heavy oils.

Chemical and Physical Characteristics

The following list of the chemical and physical characteristics of commercial rosin is given because of their bearing on the suitability of rosin for general industrial use:

Melting point;
Color;
Acid Number;
Saponification Number;
Crystallization;
Unsaponifiable Matter;
Volatile Oils;
Matter Insoluble in Gasoline;
Moisture;
Dirt.

Melting Point.—The melting point of abietic acid, the chief constituent of rosin, is generally given by authorities as above 160° C., but commercial rosin contains enough isomers of abietic acid and unsaponifiable matter to have a melting point very much lower than this. Wood rosin has always had a melting point lower than the average F, E and D grades of gum rosin, and this has been attributed to a higher content of resenes and volatile oils. Improved methods of manufacture have greatly reduced the volatile-oil content of wood rosin, but the resene content still holds the melting point lower than that of gum rosin.

On account of the importance of the melting point for many industrial uses, it has received much attention from producers of wood rosin. A minimum melting point is usually established and rigidly adhered to, unless a special low-melting-point rosin is desired and specified. While it is impossible to reach the melting point of average gum rosin by reducing the volatile constituents, still the reduction of these to a minimum by a number of wood rosin producers has improved the melting point, which is generally reported satisfactory for most uses.

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[1] Manager, Naval Stores Division, Hercules Powder Co., Wilmington, Del.

In the plants of the Hercules Powder Co. sixteen samples are taken at one-half hour intervals during each eight-hour shift, and from these a sample representing the average for each shift is made. The melting point of this average sample is then determined and recorded. For the rosin to be approved this melting point must be within the established specification limits.

Color.—The color of wood rosin is a ruby red as distinguished from the corresponding grades (F, E and D) of gum rosin, which are more of a brownish cast. This characteristic ruby color is derived mainly from the color bodies existing in the wood and extracted from it with the rosin, although heat treatment and small quantities of metallic coloring matter acquired during the extraction process may be partly responsible.

While the color bodies present cannot be eliminated without a special refining operation, they can be reduced to a limited extent by control of the manufacturing processes. Moreover, with reasonable uniformity in manufacturing conditions, the color of wood rosin can be held very close to an established standard. Daily control samples prepared by pouring the finished molten rosin into molds $\frac{7}{8}$ in. square, are taken every half hour. The laboratory labels each day's set of samples and compares them with color standards.

Acid and Saponification Numbers.—The acid and saponification numbers of wood rosin are lower than the average for gum rosin. This is largely due to the greater percentage of resenes present and also, to a lesser extent, to the nature of the resin acids themselves.

These constants are within the control of wood rosin producers only to a very limited extent, but as experience has shown that they remain practically uniform through the blending action of the process they are not the subject of daily control tests.

Crystallization.—Crystallization, as far as it has a bearing on the suitability of rosin for industrial purposes, divides itself into two major phases: crystallization in its own medium, and crystallization in solution. The first phase takes place principally during the cooling process in the barrel at the time of production and may exhibit crystal formations from isolated needles to dense opaque masses that have a melting point considerably above the boiling point of water. The second phase takes place during the manufacture or aging of certain products into which the rosin enters by solution.

If, during the cooling period of the molten rosin, conditions sufficiently unfavorable to crystallization are maintained, the formation of crystal rosin will be prevented or held down to a minimum. It can be seen, however, that the prevention of crystal rosin by this means, if the rosin has a strong tendency to crystallize, would not deter it from crystallizing when used in solution by the consumer.

The causes and control of crystal rosin have received the serious attention of wood rosin producers and, although much is to be learned in this connection, real progress has been made in the prevention of the dense opaque formation, which on account of its high melting point does not yield as readily to some industrial processes as the amorphous rosin.

It has been ascertained that there is a relation, though not a well-defined one, between the optical rotation of rosin and its relative tendency to crystallize. In general the higher the dextro specific rotation the less is this tendency developed. The Hercules plants, therefore, have made the specific rotation of rosin a standard test, one sample of each day's production being thus examined.

Unsaponifiable Matter.—Although unsaponifiable matter includes the volatile oils, in this discussion, it refers to the non-volatile constituents or resenes. Volatile oils are mentioned separately. The unsaponifiable matter of wood rosin is of somewhat higher content than the average for gum rosin, and is possibly of a softer nature. While it is largely responsible for the lower melting point and lower acid number of wood rosin, yet it appears to have valuable properties in some industries, since it possesses toughness, pliability and bonding strength.

The unsaponifiable content of wood rosin, not considering the volatile oils, is subject to control only to a limited extent without special treatment of the rosin. The nature of the process keeps it from varying to any great degree, and its estimation is, therefore, not made a daily routine test in our plants.

Volatile Oils.—Strictly speaking, the volatile oils come under the head of unsaponifiable matter, but on account of their importance they are herein listed separately. While the volatile oils are factors influencing the melting point, they sometimes have an important bearing in other respects on the suitability of wood rosin in industries that do not consider melting point of primary importance. The volatile constituents of wood rosin can be and are as a rule automatically kept down to a very small percentage by the finishing operations to obtain the required melting point. In respect to volatile oil content, wood rosin compares very favorably with the average common grades of gum rosin. We make no routine tests for volatile oil content since we consider the daily melting point tests sufficient.

Matter Insoluble in Gasoline.—There is no well-recognized method for determining the matter insoluble in gasoline, but some laboratories have adopted arbitrary standard methods for their own use. The gasoline insoluble content of wood rosin appears to be higher than in gum rosin, but sufficient work has not been done by us to date to justify conclusions.

Moisture.—While the normal moisture content of wood rosin has never been determined by us, the amount as judged from the total volatile matter is very small. Conditions can arise in the manufacture of wood rosin that will permit the presence of sufficient water to render rosin opaque, but, with the careful control which most rosin producers now have, such conditions rarely occur.

Dirt.—In the steam and solvent process, after the crude rosin solution leaves the extractors it is clarified by washing with water and is then stored in working tanks to be used as required. If any dirt or foreign material still remains it thus has ample time to settle out. The solution, therefore, as it goes to the evaporators is clean and free from all extraneous matter, which insures a clean rosin.

Summary.—Summing up the foregoing, wood rosin of good quality, as now regularly produced, is ruby red in color, and as compared with average gum rosin has a lower melting point, lower acid and saponification numbers, higher values for unsaponifiable matter and matter insoluble in gasoline, and practically the same volatile content. . It excels in uniformity of color, quality and cleanliness.

USES OF WOOD ROSIN

Wood rosin finds application for some classes of products of nearly every industry using rosin. Color being by far the most important factor in limiting its use, its suitability for any one industry depends largely on the ratio existing between the light and dark colored products manufactured. Hence, its consumption in soap is almost negligible, in paint and varnish is small, and in paper is fairly large. The relative consumption of wood rosin as compared with total rosin consumption is highest in the linoleum industry, and lower in foundry and core oil, printing ink, rosin oil, leather, matches, insulation, rubber and pitches.

The cooperation of producers and consumers of rosin in determining and developing the uses of wood rosin is steadily causing its consumption to increase.

HIDING POWER OF PIGMENTS[1]

BY R. L. HALLETT[2]

SYNOPSIS

The chief object of this paper is the further development of the hiding power of pigments and to a certain extent it is a continuation of the paper on "Hiding Power of White Pigments" presented in 1922.

The paper discusses the hiding power of pigments and the pigment characteristics which influence hiding power. It discusses rather fully the relation between tinting power and hiding power and develops a formula from which the actual hiding power of a pigment in square feet per pound may be calculated if the tinting power has been determined.

The American Society for Testing Materials defines hiding power as:[3]

> *Hiding Power.*—The power of a paint or paint material as used to obscure a surface painted with it.
>
> In this definition the word "obscure" means to render invisible or to cover up the surface so that it cannot be seen.

Hiding power is therefore an optical term and refers to the power of a paint or paint material to prevent the transmission of light through it, because if light is transmitted through a paint or paint material it may be absorbed or reflected by the surface underneath and the surface will not be obscured.

This conception of the term "hiding power" carries with it the understanding that the paint or paint material differs in color or in brightness or both, from the surface which is to be obscured, because of course there would be no observable change in the appearance of a surface if it were painted with a paint having the same color and brightness as the surface itself and it might even be considered that such paint had completely hidden the surface when only a very small amount of paint had been applied, although such hiding would be apparent and not actual as the surface underneath would still be visible from the theoretical optical point of view.

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[1] Contributed by the Research Laboratories of the National Lead Co.

[2] Chemist, National Lead Co., Brooklyn, N. Y.

[3] Standard Definitions of Terms Relating to Paint Specifications (D 16-24), 1924 Book of A.S.T.M. Standards, p. 877.

The difference in color and brightness between the paint and the surface on which it is applied may be great or it may be very small and the nearer the color and brightness of the paint approach the color and brightness of the surface painted the smaller will be the thickness of paint required apparently to obscure the surface. Konig and Brodhun[1] have shown that the least difference in brightness which is perceptible to the human eye is from one to two per cent and as the color and brightness of the paint approach the color and brightness of the surface underneath the one to two per cent perceptible difference is more quickly reached and apparent hiding is obtained with less thickness of paint.

In other words, if to a paint which differs from the surface to be painted in color and brightness we add some pigment which has the same color and brightness as the surface to be painted, the surface will be apparently hidden with a smaller thickness of this tinted paint than would be required if the original untinted paint were used.

The hiding power of a pigment as used in the form of paint is largely determined by three of its physical characteristics as follows:

1. Color and brightness;
2. Particle size or fineness;
3. Refractive index.

As already discussed the nearer the color and brightness of the paint are to the color and brightness of the surface painted, the greater will be the apparent hiding power of the paint and, of course, also of the pigment used in the paint because the paint gets its hiding power, color and brightness from the pigment.

The particle size influences the hiding power of a pigment and the paint in which the pigment is used because hiding power is a function of the specific surface of the pigment and the finer the pigment the greater will be its specific surface and the greater will be its hiding power. It must be remembered, however, that increased fineness, that is, increased specific surface, also results in greater oil-taking power and a reduction in the proportion of pigment which can be put in the paint and still maintain the necessary brushing consistency. This reduction in proportion of pigment may result in marked decrease in the hiding power of the paint after it has been spread in a practical manner even though the pigment itself may have had its hiding power increased because of its greater fineness.

The hiding power of a pigment is influenced by its refractive index or rather by the difference between its refractive index and that of the vehicle in which it is used. As most of the paint and varnish vehicles have refractive indexes very close to that of linseed oil, we may say that, practically speaking, the hiding power of a pigment is directly proportional to the difference between its refractive index and the refractive index of linseed oil.

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[1] P. G. Nutting, "Outlines of Applied Optics," p. 126 (1912).

The relationship between refractive index and hiding power of pigments explains the fact that we have so few white pigments which have any real hiding power. Practically speaking, we have but five white pigments if we consider that a pigment must have hiding or "pigmentary" power. The hiding power and tinting power of these five white pigments have been determined as follows:[1]

	Pigment	Hiding Power, sq. ft. per lb.	Hiding Power, sq. cm. per g.	Relative Hiding Power, Weight Basis	Relative Hiding Power, Volume Basis	Relative Tinting Power, Weight Basis
No. 1.	Carbonate White Lead	20	40	100	100	100
No. 2.	Basic Lead Sulfate	15	30	75	72	85
No. 3.	Titanox	40	80	200	128	350
No. 4.	Zinc Oxide	23	46	115	96	170
No. 5.	Lithopone	25	50	125	80	200

In the table the specific hiding power values refer to apparent complete hiding of a black surface over which the pigment is applied in the form of paint.

A number of other white powdered materials are sometimes erroneously called pigments, but the refractive indexes of most of them are very close to the refractive index of linseed oil and they become almost transparent where mixed with paint and varnish vehicles. Such materials are extenders and not pigments.

The American Society for Testing Materials defines tinting power or tinting strength as:[2]

> *Tinting Strength.*—The power of coloring a given quantity of paint or pigment selected as a medium standard for estimating such power.

As tinting power or tinting strength is a relative term, determination of tinting power requires a selected standard for comparison. The tinting power values for the white pigments given in the table refer to basic carbonate white lead which was selected as the standard and was given a tinting power value of 100. Good average commercial pigments were used in obtaining the data given in the table and the tinting power values of the other white pigments are based on the basic carbonate white lead standard. In determining the hiding-power and tinting-power values the pigments were mixed with bleached linseed oil because, to be of practical value the hiding-power and tinting-power determinations must be made in a vehicle having approximately the same refractive index as the vehicles which will be used with the pigments in paint and varnish mixtures.[1]

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[1] R. L. Hallett, "Hiding Power of White Pigments," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 523 (1922).

[2] Standard Definitions of Terms Relating to Paint Specifications (D 16 – 24), 1924 Book of A.S.T.M. Standards, p. 879.

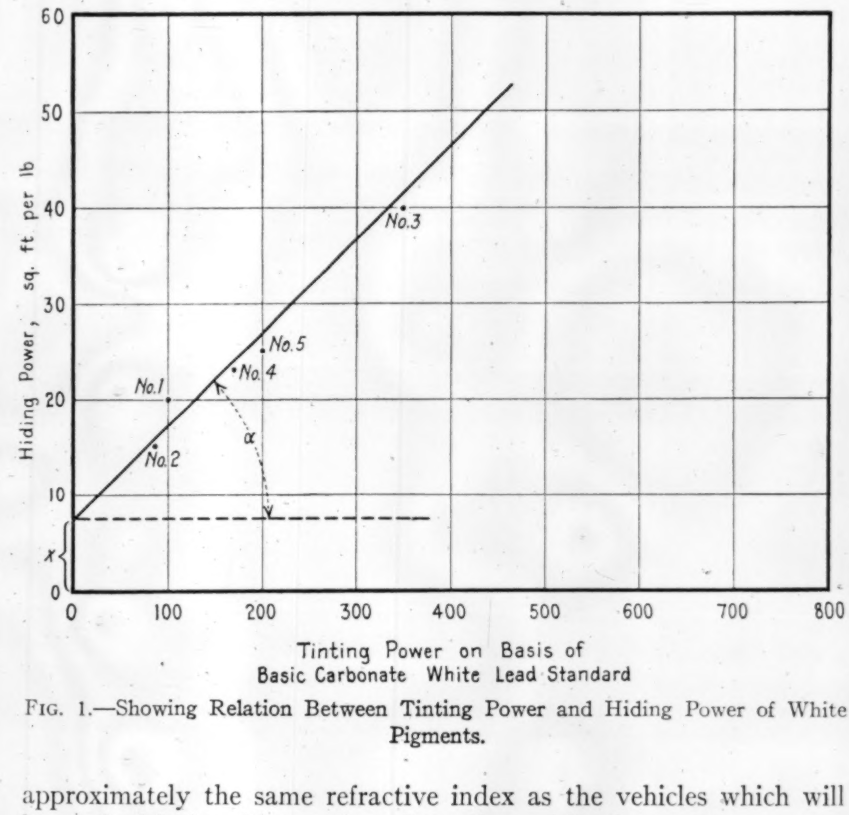

FIG. 1.—Showing Relation Between Tinting Power and Hiding Power of White Pigments.

It has been demonstrated[1] that the tinting power and hiding power of pigments are directly related and that the hiding power is directly proportional to the tinting power for pigments which have the same color and brightness. This relationship was to have been expected because particle size and refractive index, the other physical characteristics which determine hiding power, evidently also determine tinting power.

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[1] R. L. Hallett, "Hiding Power of White Pigments," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 523 (1922).

When pigments are compared which do not have the same color and brightness the hiding power will not be directly proportional to the tinting power because as the color and brightness of a pigment approach the color and brightness of the surface over which it is to be applied the apparent hiding power of the pigment is increased, as already discussed, and the tinting power is decreased because changing the color and brightness to resemble more nearly the surface to be painted is really the same as introducing pigment which would be added as the medium standard in determining the tinting power and of course less of the medium standard could then be added in the tinting power test. This of course assumes that the medium standard used in the tinting power test is more like the surface to be painted in color and brightness than is the pigment which is being tested.

If the hiding power values expressed in square feet per pound and the tinting power values of the white pigments given in the table are plotted on squared paper using the hiding power values as one coordinate and the tinting power values as the other, a straight line is obtained when the plotted points are connected as shown in Fig. 1. The points are numbered to correspond to the pigments given in the table. In this plot the plotted hiding power units are ten times the length of the plotted tinting power units.

The points do not lie exactly on the straight line because the points are obtained from experimental work in which there doubtless were some experimental errors, whereas the line is drawn so as to occupy its calculated position as described further on in this paper. From this plot it will be seen that the straight line drawn through the plotted points, when extended toward the vertical hiding-power axis, intercepts the hiding-power axis at some point above the horizontal tinting-power axis, which indicates that, for pigments having the same color and brightness as those used for making the plot in Fig. 1, when the limit is reached and such a pigment has no tinting power it will still have some hiding power. This is probably due to the fact that paints made with these pigments have considerably less than 100 per cent brightness and absorb a considerable part of the light which strikes them. The appreciable proportion of light which is absorbed probably influences the hiding power and gives some hiding power to such a paint even when it has no tinting power.

The length of the intercept on the vertical hiding power coordinate may be determined by calculation as follows:

Let x = the length of the intercept in square feet per pound;

a = the angle formed by the plotted straight line and the horizontal;

H = hiding power (square feet per pound) of any point on the plotted straight line; and

T = the corresponding tinting power value for the same point.

Then,

$$\tan \alpha = \frac{10(H-x)}{T} \quad \text{.................} \quad (1)$$

Inserting in Eq. 1 the values of H and T for the five white pigments given in the table gives five different $\frac{10(H-x)}{T}$ expressions each equal to tan α and therefore each equal to each of the others. Equating each $\frac{10(H-x)}{T}$ expression with each of the others and solving each equation so obtained for x we have ten values for x, the average of which is 7.5. Therefore $x = 7.5$.

Inserting in Eq. 1 the value of x and the values for H and T for the five white pigments given in the table gives five different equations and solving each equation for tan α gives five different values for tan α, the average being 0.97. Therefore $\tan \alpha = 0.97$ and $\alpha = 44$ deg. (approximately).

Inserting in Eq. 1 the values for x and tan α we have $0.97 = \frac{10(H-7.5)}{T}$ from which we have the following:

$$H = 0.097\, T + 7.5 \quad \text{.................} \quad (2)$$

That is, the hiding power of any white pigment in square feet per pound is equal to its tinting power multiplied by 0.097, plus 7.5.

Equation 2 applies to all white pigments having approximately the same brightness as those given in the table when hiding is considered as the obscuring of a black surface by the white pigment mixed with linseed oil in the form of paint.

Tinting power may be determined by comparison with any selected medium standard which may in turn be calculated to the basic carbonate white lead standard and the result may then be used directly in Eq. 2.

DISCUSSION

Mr. Breyer. MR. F. G. BREYER.[1]—There is an important statement in this paper that persists all through the literature, and Mr. Hallett is no more guilty than a lot of others in passing it on without scrutiny. The statement is that the finer the pigment the greater will be its specific surface and the *greater will be its hiding power.* I can illustrate the fallacy of that statement very clearly by pointing out to you a simple phenomenon that we all know. Take raindrops, for example; the hiding power of a good heavy rainstorm is very low because the raindrops are quite coarse. Take the same amount of water and subdivide it down to a fog and you get good hiding power, good obscuring power. Further reduce that water to molecular size, that is, down to the condition of water in this atmosphere, and it again ceases to have hiding power. The statement is not correct that the finer the pigment the greater will be its hiding power. Fineness has a definite minimum for maximum hiding power. For example, for zinc oxide, if I remember the figure rightly, it is around 0.3 micron. At that point zinc oxide has its highest hiding power, below that the individual particles in increasing degree cease to refract the light waves and the hiding power drops off correspondingly. We make an oxide of average particle size about 0.15 micron whose hiding power is very low. Some of the paint men here are familiar with it. It is sold to the rubber people because of its extraordinary fineness, large surface and its quick chemical reactivity.

Another point in Mr. Hallett's paper is that hiding power is a function of the specific surface. It seems to me a better way to think about this phenomenon is as follows: Hiding power is proportional to the number of entries and exits from media of one refractive index into media of another refractive index. A little light is reflected at each surface of entry and a little at each surface of exit. Of course, the greater the difference in refractive index between the two media, the more light is reflected at each entry or exit and the less number of particles it can pass through before it is all reflected. The essential requirement of white, or approximate white, is two transparent materials of different refractive index, one dispersed in a finely divided condition, but not too fine, in the other. Fine droplets of water in air (*i. e.*, freshly condensed steam) give white equally as well as fine droplets of air in water (foam). **Mr. Breyer.**

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[1] Chief, Research Division, New Jersey Zinc Co., Palmerton, Pa.

Mr. Hallett. MR. R. L. HALLETT.—I do not think the criticism of my statement that the hiding power is a function of the fineness applies to this paper and in this connection. There is no question but what hiding power is influenced by the fineness, and increases with the fineness. We all know that is one of the basic principles of the manufacture of pigments and the manufacture of paint. From the practical standpoint of the manufacture of pigments, and this is intended to be a practical paper, there is no question but what the size of the particle is very important in the estimation of hiding power. Furthermore, I believe that the expression "specific surface," concisely and in the fewest words, carries the thought that I wanted to convey.

THE STUDY OF NITROCELLULOSE LACQUERS BY THE STRESS-STRAIN METHOD[1]

By G. W. Rundle[2] and W. C. Norris[2]

Synopsis

Films of nitrocellulose lacquers are easily obtained for testing purposes by flowing them on amalgamated tinned sheet-iron panels, setting them up at an angle of about 15 deg. from the vertical and allowing the excess material to flow off. The specimen-cutting die and the tension testing machine are the same as those used for testing films of oleoresinous materials.

The method of testing the film specimens is mentioned. Factors influencing results such as thickness of film, humidity and temperature are discussed.

The following variations of lacquer composition are taken up from the stress-strain point of view: Plasticizers and solvents are first tested singly and in various combinations, with and without heat treatment of the film. Resins are then taken up, films being measured under both desiccated and high humidity conditions. Finally the effects of various pigments are shown.

Introduction

Whatever effect other factors may have upon the life or period of useful service of a nitrocellulose lacquer film, it is a well-recognized fact that its retention of distensibility within certain limits is a correct measure of its life. At least, the rate at which this property changes gives an indication as to the stability of the system. This property is directly or indirectly determined by almost everyone concerned with lacquers. The methods vary from the simple one of flowing out on a piece of metal and bending over a mandrel to the slightly more involved, but certainly more scientific and comprehensive, method of complete stress-strain determinations.

An apparatus that determines and records the stress-strain characteristics of lacquer films gives a more or less complete picture (from the curves) of the behavior and actual functioning of the various ingredients. For instance, it will determine whether a high boiling material is merely a film softener or a true plasticizer imparting desirable distensible properties to the film.

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[1] A contribution from the Research Laboratories of The New Jersey Zinc Co.

[2] Investigators, Paint Section, Research Division, New Jersey Zinc Co., Palmerton, Pa.

It is the purpose of this paper to point out certain factors involved in the stress-strain measurement on lacquer films, to present certain typical curves and data obtained, and to interpret these curves and data.

PREPARATION OF FILM SPECIMENS

Films are prepared by flowing the nitrocellulose lacquers or lacquer enamels on amalgamated tinned sheet iron and setting them up, at an angle of about 15 deg. from the vertical, on a blotter or other absorbent material. Generally, flowing rather than spraying is to be preferred because of greater ease in maintaining uniform film thickness. After hardening for a few hours they are stripped, cut to test specimens with a suitable die, and the thickness measured for classification purposes. Hard, brittle films must usually be stripped within an hour or even less.

The film specimens are placed in a well-ventilated cabinet for aging. Two or three days prior to testing they should be placed in a humidor maintained at a known and constant relative humidity. A humidor containing the correct percentage (43 per cent) of H_2SO_4[1] to maintain a relative humidity of 50 per cent at room temperature is suitable.

APPARATUS

The machine used in these measurements is the same, with a few improvements, as that described elsewhere in the Proceedings of the Society.[2] A more recent and complete description is to be found in a research bulletin issued by The New Jersey Zinc Co.[3] Heavier springs are the only additional equipment required over that used in the measurement of oleoresinous films. Due to the high tensile strength of nitrocellulose lacquer films, as compared with oleoresinous films, it is necessary to use a higher loading rate. For the average lacquer film a convenient loading rate is 50 kg. per sq. cm. per minute as compared with 15 kg. per sq. cm. per minute for oleoresinous materials.

TESTING THE FILM SPECIMENS

The film to be measured is taken from the humidor and tested immediately. Since it takes only a minute to test the average lacquer film no appreciable change in the moisture content can take place.

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[1] Robert E. Wilson, "Aqueous Vapor Pressure and Density of Sulfuric Acid Solution as a Function of Temperature and Composition," *Journal of Industrial Engineers*, Vol. XIII, No. 4, 1921.

[2] H. A. Nelson, "Stress-Strain Measurements on Films of Drying Oils, Paints and Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1111 (1921).

[3] H. A. Nelson and G. W. Rundle, "Some Physical Properties of Paint and Varnish Films."

Furthermore, most lacquer films are not susceptible to marked changes in physical properties with variations in humidity, as will be pointed out later. Enough specimens should be measured to obtain check results. Five are usually sufficient, except on particularly hard and brittle films. It is usually the case that the curves coincide but vary more or less as to the ultimate breaking point. This is due, generally, to unavoidable minute flaws along the edges of the test specimen. It is permissible in this case to choose the curve which shows the greatest load.

INFLUENCE OF TEMPERATURE AND HUMIDITY

The effect of temperature on the distensible qualities of nitrocellulose lacquers is, in general, greater than on oleoresinous materials. It is advisable to maintain the temperature of the room in which the measurements are made within as narrow limits as possible.

The influence of humidity is, in most cases, less in nitrocellulose lacquers than on oleoresinous materials. The hygroscopicity of the constituents is, of course, the most important factor. The kind and amount of gums used exert a considerable influence. The effect of humidity in a few different lacquers containing various resins is pointed out in the curves of Fig. 5.

INFLUENCE OF FILM THICKNESS

As mentioned above, the specimens are classified according to average thickness when they are cut. It is desirable to compare only films of nearly the same thickness for two reasons.

The first reason is one of expediency only. To maintain a constant loading rate with films differing widely in thickness, it is necessary to use springs of different values and adjust the speed. It is obviously much more convenient to use one spring and slightly varying speeds throughout a given series of tests.

The second reason for using films of approximately the same thickness is because of retention of volatile matter by a film. This has been found to be quite an important factor in the measurements of lacquer films that have aged for less than a week or for even longer periods in the case of certain solvent compositions. The two main factors in the degree of volatile retention are film thickness and nature of solvents used. Considering a series of the same solvent composition the films should be of approximately the same thickness if tests are to be run within a week or two after flowing.

The curves shown in Fig. 1 clearly illustrate the erroneous conclusions one is liable to draw when comparing films differing widely in thickness. It is true that the solvent mixture used serves to exaggerate this effect. Nevertheless, the advisability of comparing

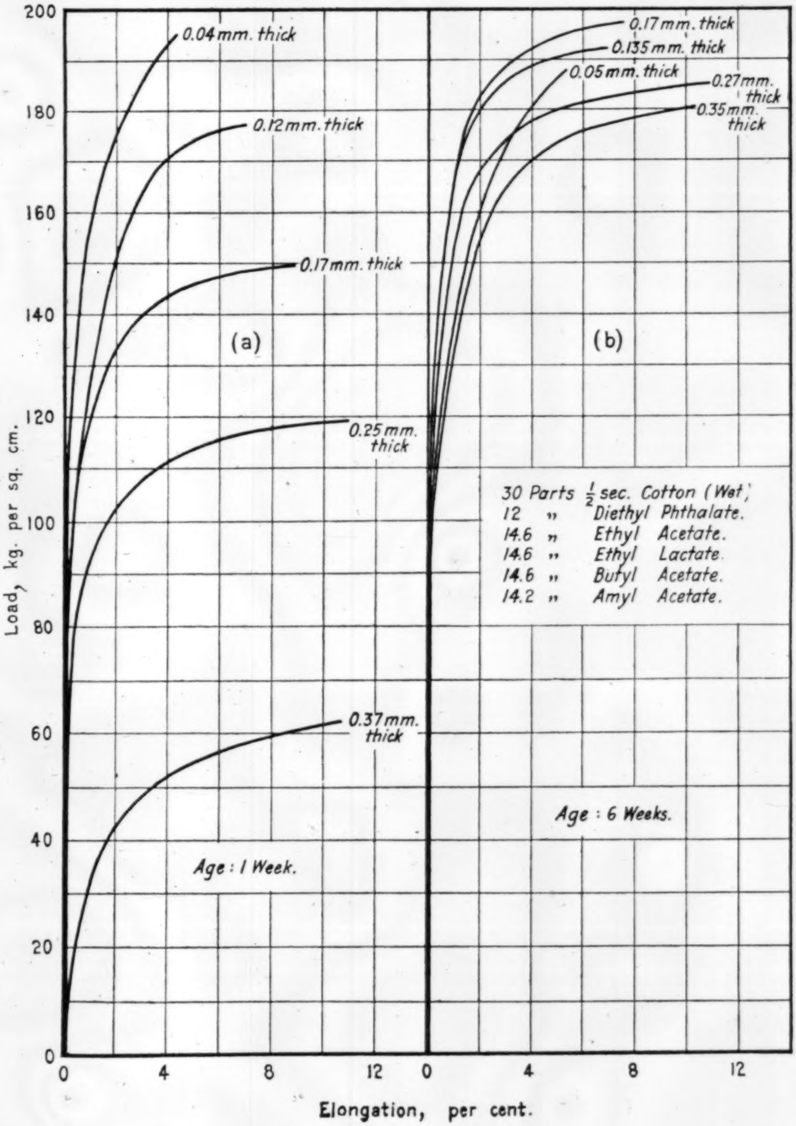

FIG. 1.—Influence of Film Thickness on the Retention of Volatile Matter.

stress-strain measurements on films of approximately the same thickness, particularly in the early stages of aging, is apparent.

PLASTICIZERS AND SOLVENTS

Figure 2 represents curves obtained at different agings of films made up from the same base formula, the plasticizer and solvent content being varied as indicated. (Parts given in this formula and all succeeding ones are by weight.) The films all hardened progressively with age, the true plasticizers with the higher boiling points changing

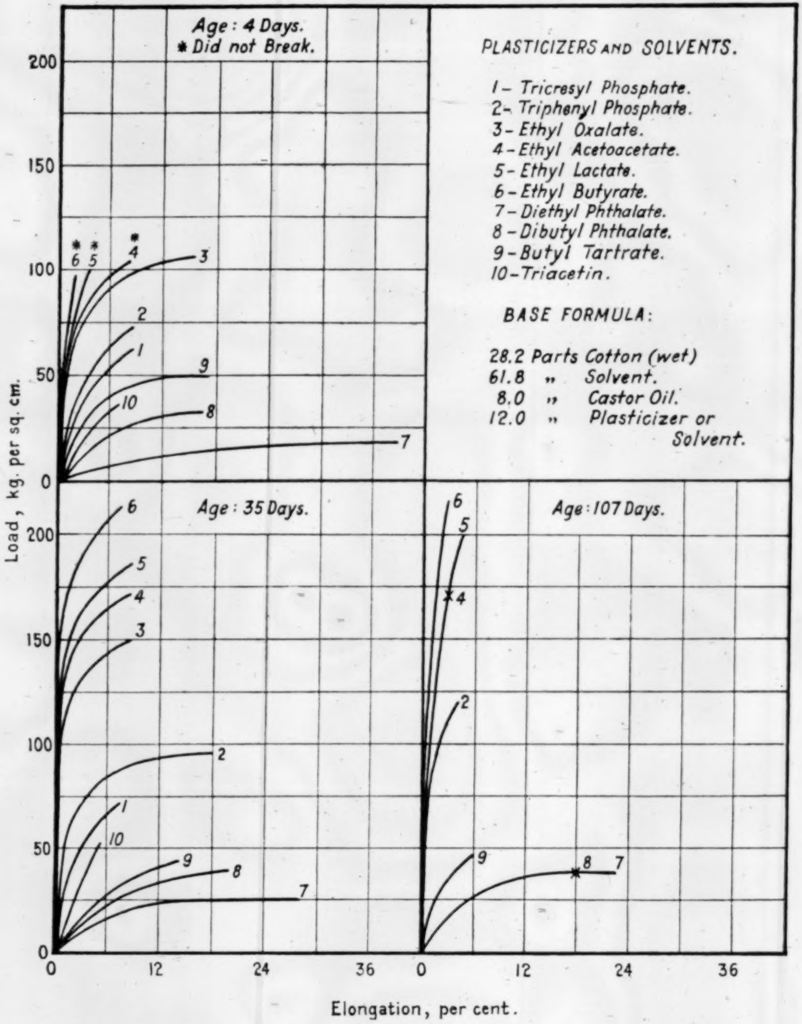

FIG. 2.—Characteristics Imparted by Various Plasticizers and Solvents.

the least. The positions of the curves indicate the relative plasticizing power of the materials used, at the time the films were tested. There was an insufficient number of films to measure Nos. 1, 3 and 10 at 107 days' aging; also an insufficient number of all films to continue the study or we might expect, from the curves shown in Fig. 3, that they would ultimately be in different relative positions.

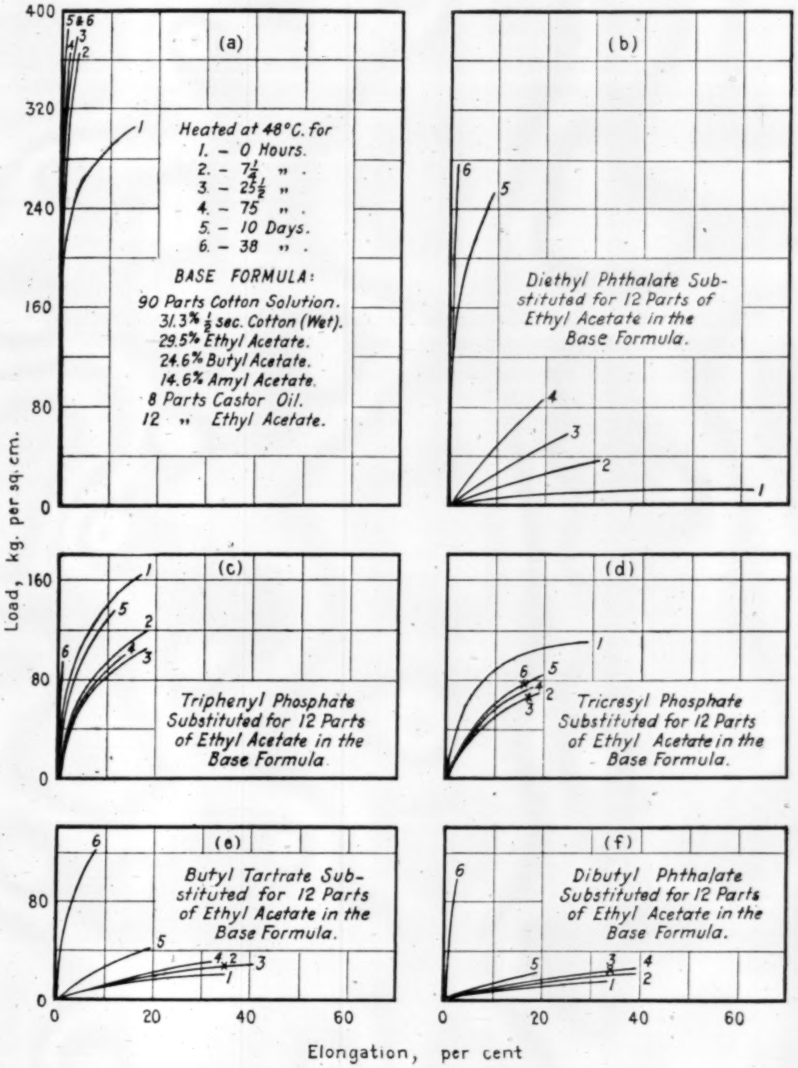

FIG. 3.—The Effect of Slightly Accelerated Aging on a Few Plasticizers.

Figure 3 represents measurements of six films made from the same base formula but each containing a different plasticizer. The test specimens cut from these films were heated in an oven at 48° C. for the time noted in Fig. 3 (*a*). The films were taken from the oven

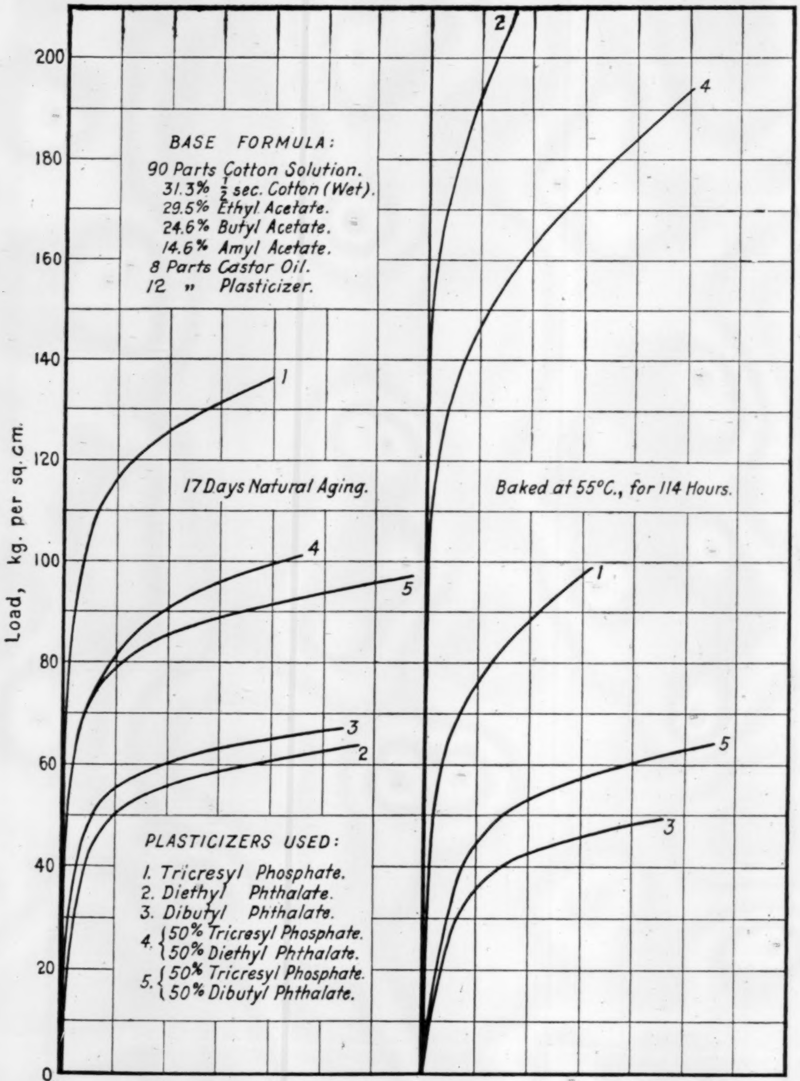

FIG. 4.—Plasticizers Used Singly and in Combinations.

and allowed to come to room temperature before testing. Identical curves were obtained whether the heated films were allowed 15 minutes or 24 hours to come to room conditions.

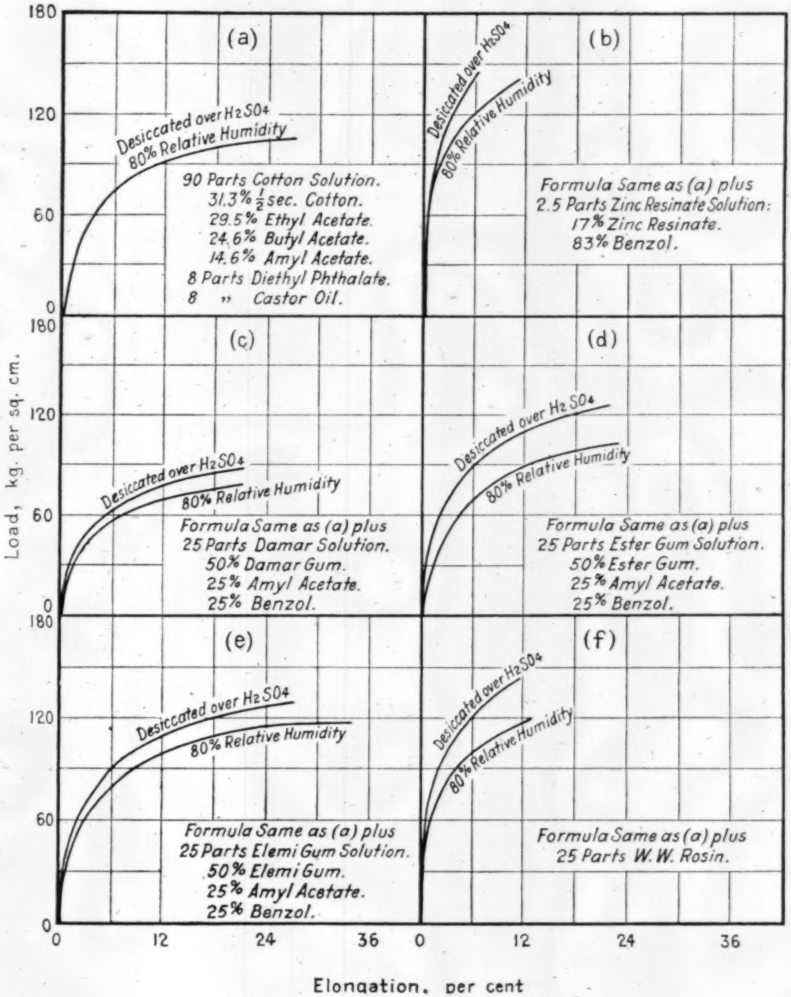

FIG. 5.—Characteristics Imparted by Various Resins.

The films containing the phosphates as plasticizers (*c*) and (*d*), softened at first on heating then became harder, while those containing the phthalates (*b*) and (*f*), butyl tartrate (*e*), and the one with no plasticizer (*a*), hardened progressively from the beginning. After 38 days' heating the film containing tricresyl phosphate was the softest, softer even than the original that had no heat treatment. The film containing triphenyl phosphate became very brittle with practically no elongation. This contrast in the behavior of triphenyl and tricresyl phosphates has been mentioned and discussed by Nelson and McKim.[1]

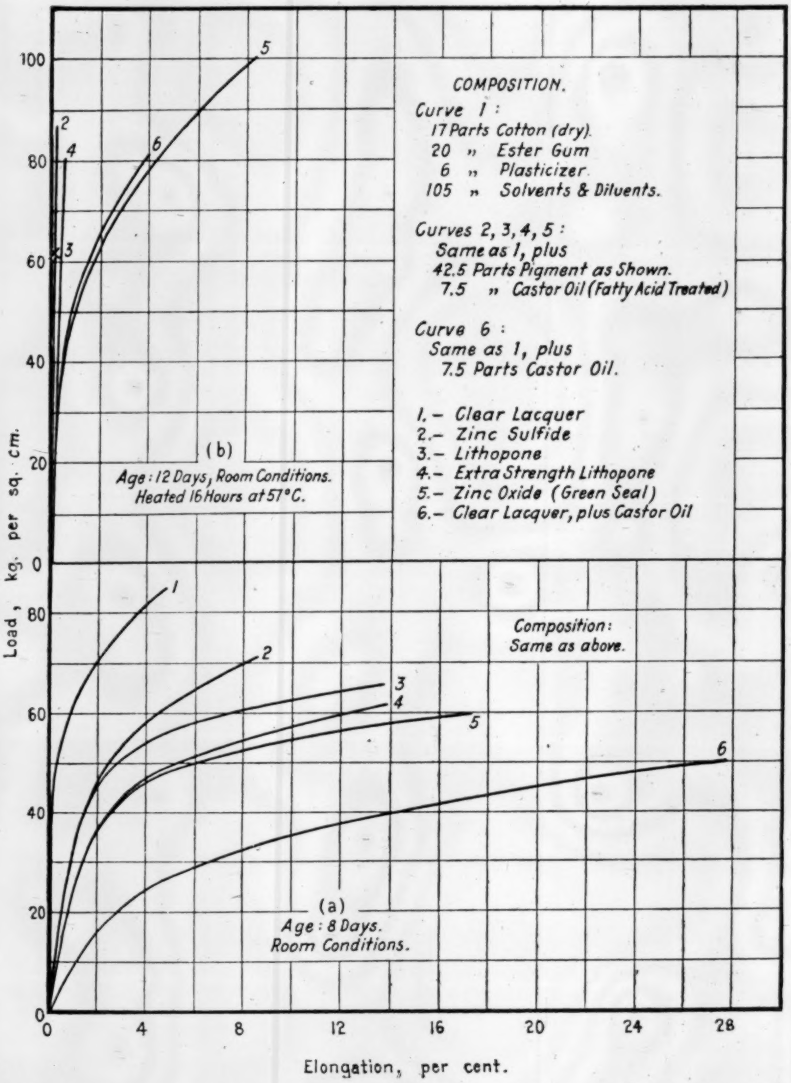

FIG. 6.—Characteristics Imparted by Four Different Pigments Under Slightly Accelerated Aging Conditions.

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[1] *Paints, Oils and Drugs*, January, 1926; and *Canadian Chemistry and Metallurgy*, January, 1926.

Figure 4 is a typical illustration of the use of stress-strain determinations in observing the effect of various combinations. The extreme range of changes in the physical properties of the film plasticized with diethyl phthalate alone is to be noted. At 17 days it is the softest film and after aging with heat (55° C.) becomes the most brittle. From a study of the curves it is apparent that under slightly accelerated aging conditions the phthalates carry over more of their individual characteristics, when used in combination with tricresyl phosphate, than does the latter.

RESINS

Figure 5 illustrates the effects imparted by five different resins. It will be noticed that the results on films containing no resin, Fig. 5 (*a*), are the same at 80-per-cent relative humidity as those desiccated. When, however, resins are introduced the film becomes more sensitive to moisture depending upon the kind and amount used.

In another series of curves (not shown) it was found that the acid number of ester gum was not a factor in film strength when the acid number of the gum varied from 7 to 20. In this series the specimens were aged for nine months and contained 13 parts of gum to 21 parts of dry cotton.

PIGMENTS

Figure 6 is a series representing the effect of equal weights of four representative pigments upon the distensible properties of a film. For comparison purposes two other curves are shown, the one being the clear lacquer, the other the clear lacquer plus the amount of castor oil introduced by the pigment pastes in the four pigmented films. The curves in Fig. 6 (*b*) represent measurements taken after aging of the films at 57° C. for 16 hours. Under these conditions the clear lacquer film, No. 1, became too brittle for measurement.

The most striking feature of the curves is the fact that the incorporation of zinc oxide seems materially to extend the distensible life of the lacquer as reasoned from the curves. It is a recognized fact that the incorporation of zinc oxide in a clear lacquer will increase its life when exposed to ultraviolet light. However, it is to be remembered that in these experiments the films were not exposed except to ordinary room light conditions, so that the beneficial effect must be ascribed to some other cause. It might possibly be ascribed to a stabilizing action due to a ready neutralization of acid decomposition by-products, which are known to accelerate the decomposition of nitrocellulose, especially under slight heating.

MECHANICAL TESTING AND RECORDING OF THE DRYING OF PAINTS AND VARNISHES

By John McE. Sanderson[1]

Synopsis

The progress of drying or solidification of a paint or varnish film may be divided into three periods. Variations in these three periods affect the practical working qualities of the material. The paper describes a mechanical device for testing and recording the rate of drying through these three periods and describes improvements in the drying-time meter covered by the author in a previous paper.[2]

Drying is defined by the society as "The solidification of a film."[3] This definition is rough and perhaps inaccurate in that most paint films in their original condition are not true liquids and the dried film does not generally reach the state of true solidity. The term "drying" is, however, generally accepted as covering the change from a material of comparatively low viscosity and a rather high degree of plasticity to a material of sufficiently low plasticity so that it will not flow under ordinary handling. After the film is dry, there is a further hardening with age but the solidified film remains more or less plastic.

During the process of drying, there are no sharp points of change, but there are three fairly well defined transition periods which determine the practical working qualities of the material. These three periods are as follows:

1. Initial Set when the material reaches a degree of viscosity and plasticity beyond which it cannot be worked. If applied with a brush, permanent marks of the bristles will remain or if sprayed or dipped, it will no longer flow out properly.
2. Surface Drying when dust will not readily adhere or when the film may be touched lightly without its feeling sticky. This is the point by which the drying of a coating is usually judged. Coatings intended for drying under ordinary conditions of temperature, etc., must reach this degree of drying within a comparatively few hours to avoid damage to the coating from dust, rain, insects or casual handling.

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[1] Manager, Larkin Co., Inc., Paint Division, Buffalo, N. Y.

[2] J. McE. Sanderson, "A Recording Drying-Time Meter for Varnishes and Similar Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 407 (1925).

[3] Standard Definitions of Terms Relating to Paint Specifications (D 16 - 24), 1924 Book of A.S.T.M. Standards, p. 879.

3. Drying Hard when the film has become solid all the way through with a plasticity so low that it can be subjected to ordinary handling without marring. Many finishes are judged for hardness of drying by the time required for the film to reach a point at which it can be rubbed smooth with pumice. Some finishes are judged for hardness by the time required to reach such a low degree of plasticity that the film will not "print," that is, will not show permanent marks from a weight applied to a small area usually at an elevated temperature.

To correctly indicate the drying properties of a given material, it should be tested in such a manner as to record the limitations of the three transition periods indicated above. It has been thoroughly demonstrated by the work of Sub-Committee IX on Varnish of Committee D-1 on Preservative Coatings for Structural Materials that the method in common use for drying tests, namely, touching the film at intervals with the finger, is extremely inaccurate because of the wide variation in results obtained by different operators working on the same material under the same conditions.[1] To eliminate the effect of the personal equation, we are seeking, therefore, to devise a mechanical means of testing which will record automatically the gradual changes taking place in a drying film.

The writer described last year,[2] a mechanical device for this purpose. In it the material was coated by centrifugal force on a flat disk which was then revolved by a clock mechanism so that a stream of sand flowed onto the film in a spiral path. When the test was completed and the excess sand brushed off, that which remained adhering to the film indicated the time required for the film to dry on the surface. Further work with this mechanism during the past year has confirmed the results prevously reported, and has indicated that this mechanism gives a satisfactory method of testing and recording the second transition period in a drying film, namely, the time of drying on the surface.

While the essential features of this mechanism remain the same as previously described, the work during the past year has resulted in some changes and improvements in the design. The mechanism which combined coating and testing features proved to be quite complicated and expensive to construct and failed to give the expected advantages.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part I, p. 381 (1922); also, Report of Sub-Committee IX on Varnish of Committee D-1 on Preservative Coatings for Structural Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I p. 324 (1926).

[2] J. McE. Sanderson, "A Recording Drying-Time Meter for Varnishes and Similar Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 407 (1925).

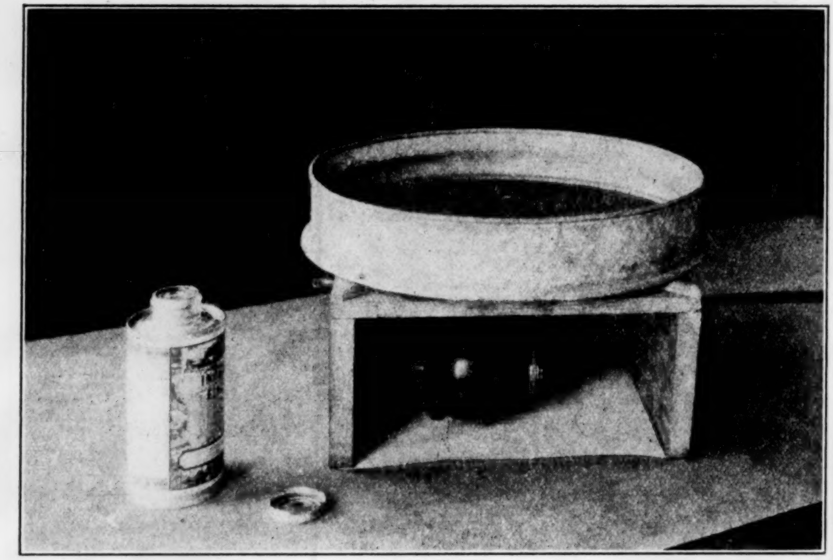

FIG. 1.—Spinning Mechanism Used for Coating Disks with Material to be Tested.

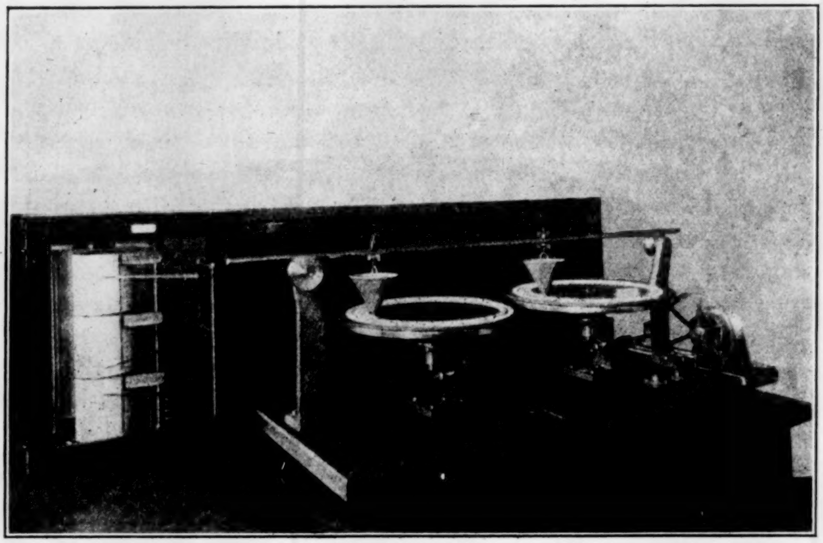

FIG. 2.—Drying-Time Meter Arranged for Test of Surface Drying Only. Instrument at Left Records Temperature, Humidity, and Atmospheric Pressure.

It was, therefore, found desirable to return to the original design wherein separate mechanisms were used for coating and testing.

The mechanism for coating (shown in Fig. 1) consists of a phonograph table rotated in a horizontal position by a small electric motor equipped with a governor to permit close regulation of the speed. Around the table is a pan to catch the excess varnish thrown off the disk in the process of coating. We have found it desirable to reduce the speed at which the disks are revolved while coating to 265 r.p.m. instead of 350 r.p.m. as used in previous tests. The slower speed appears to give a film more nearly commensurate with that which will be obtained with the same material under ordinary methods of application. This is highly desirable for all drying tests, and particularly for tests on initial set which are very misleading when the film is too thin.

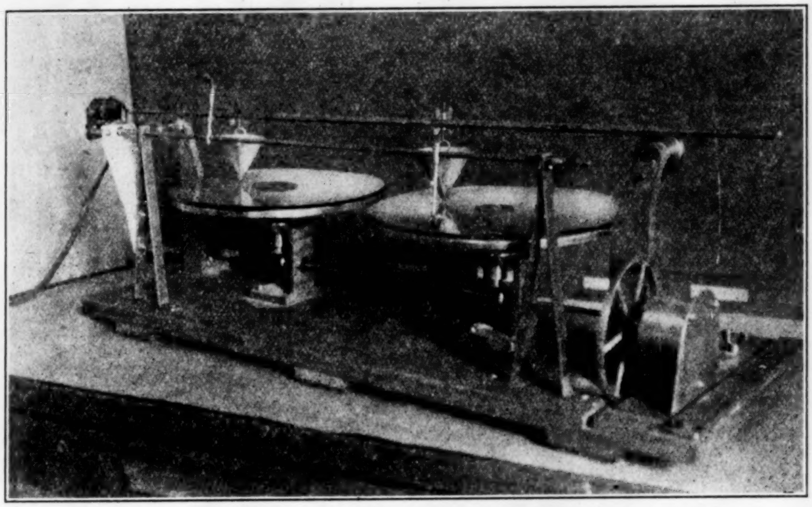

FIG. 3.—Drying-Time Meter Complete with Attachment for Testing Time of Initial Set.

The design of the mechanism for testing has been simplified as far as possible. Figure 2 shows this machine arranged for comparative testing of two materials although the same design has been carried out with three and four disks, and machines are being tried out with a still greater number. For the variety of materials which it is desirable to test on this mechanism, ranging from quick drying varnishes to slow drying oils, a range of three speeds of the disks appears sufficient. These speeds, one revolution in three hours, once in twelve hours, and once in twenty-four hours, are all obtained from the same clock movement by a change of gears made before the test is started. A fourth speed of one revolution per hour or less has been included in some tests to sharply differentiate quick drying materials. To satisfactorily obtain this speed, however, the use of a separate clock mechanism is required, and for the ordinary run of materials, the three speeds mentioned above have been found sufficient.

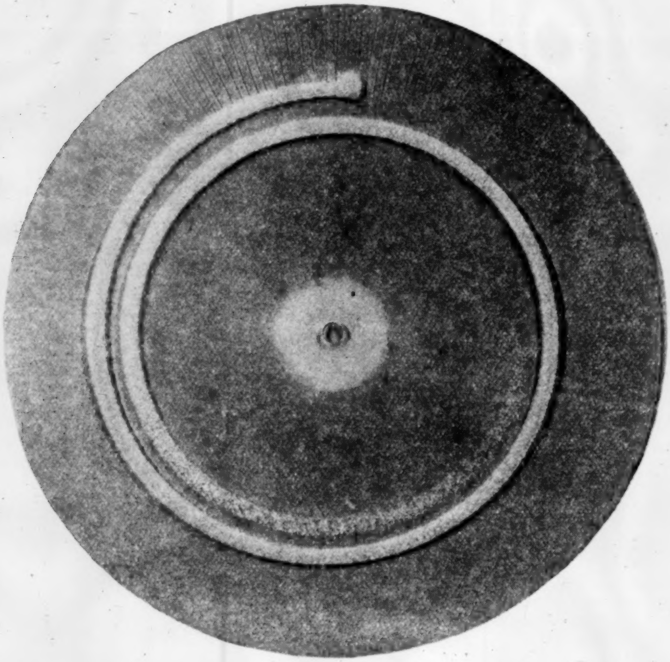

FIG. 4.—Typical Tests Showing Record of Time of Initial Set and Time of Surface Drying.

The drying-time meter as described above, while giving a very satisfactory indication of the time required for a film to dry on the surface, gives no indication of the time required for initial set nor for the film to dry hard. Our recent work has been an effort to adapt the instrument to the testing and recording of these other two periods.

If a pin point is drawn rapidly across a wet film, the flowing out of the mark which it leaves gives a fairly accurate indication of how the material will flow out when brushed, as the pin point acts in a manner quite similar to the individual bristles in a brush.[1] As long as the mark left by the pin point will flow out and disappear, the material can still be brushed without showing permanent brush marks. When the film has set up so that marks from the pin point remain permanently, it has also reached the stage where it will no longer flow out when brushed, sprayed, or dipped.

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[1] A. H. Pfund, "Tests for Hardness, Gloss, Color and Leveling of Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 392 (1925).

Having determined that this pin scratch method offered a satisfactory means of testing initial set, it was comparatively simple to devise an attachment for the drying-time meter previously described which would test and record the initial set. Figure 3 shows the drying-time meter complete with this attachment. A shaft mounted horizontally above the outer edge of the coated disks is revolved by a separate mechanism at a speed of one revolution per minute. Carried on this shaft are radial arms terminating in steel points arranged so as to press against the coated disk for a distance of about $1\frac{1}{2}$ in. from the outer edge. When this test is carried out with the disks rotating at a speed of once in three hours, the marks made by the steel points are about 0.20 in. apart. When this test has been run to completion, that is, after several marks in succession show no indication of disappearing, the mechanism carrying the points is stopped, the funnels are placed in position, filled with sand, and the testing of the time of surface drying carried out on the same disk. When the material is thoroughly dry, the test is stopped, and the disks brushed off as

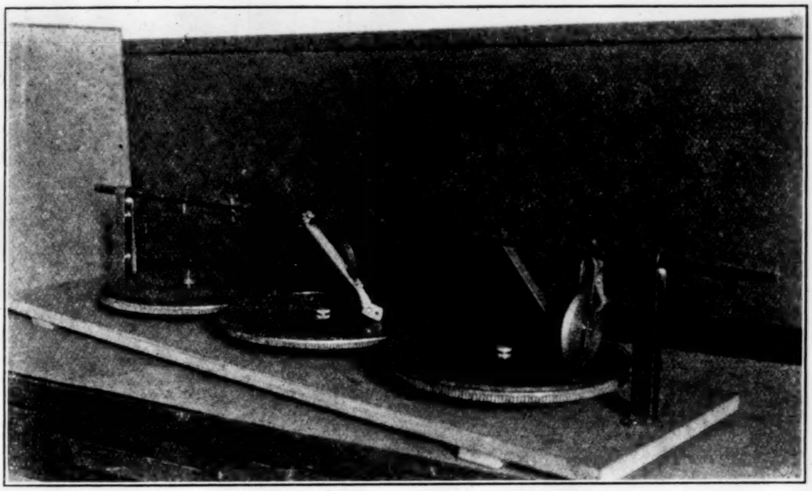

FIG. 5.—Drying-Time Meter Equipped with Two Different Experimental Devices for Determination of Time of Drying Hard.

usual. The appearance of a disk, after the completion of such a test, is shown in Fig. 4.

The determination of the time of drying-hard has presented a more difficult problem than the determination of either of the other two transition periods. We have experimented along two different lines, and while neither of our methods has given very satisfactory results, mention of them may be justified as suggestions to other workers on this problem. The first method was to roll a heavy wheel on the film as it dried and attempt to judge the hardness by the impression which it left. The other was to cut the drying film with a razor blade held at an angle and attempt to judge the hardness by a microscopic examination of the edge of the cut. The latter method has given results more promising than the former, but has not been worked out to a point where it can be depended upon to indicate comparative hardness between different films. Figure 5 shows a drying-time meter equipped with devices for testing hardness according to the two methods described above.

The drying-time meter described is adapted to comparative tests only. Results obtained on duplicate tests on the same material at the same time check closely. In order to duplicate results at different times and different localities, it will be necessary to regulate and standardize on all the conditions such as heat, light, ventilation, etc., which affect drying. It gives a means of study of the effect of various conditions which affect drying which we have not previously had and seems to be the first step toward standardizing drying tests so that we can check a batch of material for drying against an absolute standard of results recorded some time in the past instead of having to make each test against a standard sample.

The machines described above are available to any laboratory interested in drying tests. Several of them are in regular use, and it is expected that many more of them will be in use in the near future.

ACCELERATED WEATHERING: FURTHER DEVELOPMENT OF APPARATUS AND EXPOSURE CYCLES

By H. A. Nelson,[1] F. C. Schmutz[1] and D. L. Gamble[1]

Synopsis

Conclusions from the study of accelerated weathering have indicated that a flexible exposure system is desirable for obtaining the most satisfactory results. The equipment described in this paper is designed to obtain the maximum of flexibility and still be practical for routine testing. The different types of exposure cycles thus made possible are limited only by the number of practical combinations that can be made of the following:

1. Light (Mercury or Carbon Arc)
    - (*a*) At controlled, slightly elevated temperatures.
    - (*b*) With 100 per cent relative humidity.
    - (*c*) With less than 5 per cent relative humidity.
    - (*d*) In an atmosphere charged with active gases as, for example, oxygen.
2. Water Spray.
3. Refrigeration (Temperature Changes).

The introduction of silica gel as a dehydrator, which provides for humidity control without necessitating air conditioning by refrigeration, is an important advance.

The use of additional oxygen in the light exposure chamber has facilitated the reproduction of the checking type of failure.

A system of reasoning has been suggested, which can be followed out in developing an accelerated exposure cycle for any given section of the country from the available climatological data.

The decrease in the intensity of the quartz mercury arc with use varies the effectiveness of the accelerated test. Data is presented showing the voltage or amperage increases required to maintain a satisfactorily constant total intensity of the 30-in. burner as the transparency of the quartz decreases.

In view of the increasing interest in accelerated weathering as an endurance test for paint, varnish and lacquer surfaces, the basic factors governing this system of testing are gradually being analyzed and understood. The authors, in their previous work[2] on the subject, have summarized some of these fundamental factors. The next question is how this information might best be adapted for practical application as regards apparatus and manipulation in daily routine. Toward this end the authors wish, particularly, to present a design of apparatus that has been used in this laboratory which is very flexible in its applications. This apparatus is not put forward as the last word in engineering design for accelerating weathering equipment, but rather as a model to illustrate how certain manipulations fundamental to such a testing scheme can be carried out in practice.

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[1] Investigators, Paint Section, Research Division, New Jersey Zinc Co., Palmerton, Pa.

[2] H. A. Nelson, "Accelerated Weathering of Paints on Wood and Metal Surfaces," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 485 (1922); H. A. Nelson and F. C. Schmutz, "Further Study of Accelerated Weathering: Effect of Variations in Exposure Cycle Combinations on Common Types of Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 920 (1924); H. A. Nelson, Chapter IX, "Physical and Chemical Examination of Paints, etc." by H. A. Gardner—1925 Edition; H. A. Nelson and W. A. McKim, "Accelerated Weathering as Applied to Lacquer Enamels," *Drugs, Oils and Paints*, January, 1926, also *Canadian Chemistry and Metallurgy*, January, 1926; H. A. Nelson and F. C. Schmutz, "Accelerated Weathering—A Consideration of Some Fundamentals Governing Its Applications," *Industrial and Engineering Chemistry*, Vol. 18, No. 12 (December, 1926).

In addition, a general formula is suggested for deriving exposure cycles to simulate various climatic conditions, and some data are presented on the deterioration of mercury arc burners while in service.

DESCRIPTION OF APPARATUS

In line with the conclusions from previous work and, also, from a study of characteristic climatic conditions, any system, to attain the maximum of useful flexibility, should be able to approximate the following conditions:

1. Destructive light action under very humid conditions at a moderately elevated temperature;
2. Destructive light action under very dry conditions at a moderately elevated temperature;
3. Actual washing action of a water spray;
4. Temperature changes by exposure to low temperature conditions, preferably below 32° F.;
5. For simulating some very moist, warm climates, periodic wetting while under light exposure is desirable;
6. In a few instances some form of industrial gas exposure (SO_2, CO_2, etc.) is helpful in approximating actual outdoor conditions, but this form of specialized test will be disregarded at this time because of limited space.

We have been quite successful in reproducing these conditions at will by means of the apparatus shown in Figs. 1 and 2.

Exposure Chamber (Fig. 1).—The exposure chamber proper is of No. 16 gage galvanized iron, 4 ft. 2 in. in diameter, with a clear inside working height of 16 in. Taking into consideration the factors of light effectiveness, heat control and capacity, 2 ft. was chosen as being a desirable distance of the light source from the panels. A tank of this size has a capacity ranging from twenty-seven 6 by 12-in. panels to seventy-two 4 by 6-in. panels.

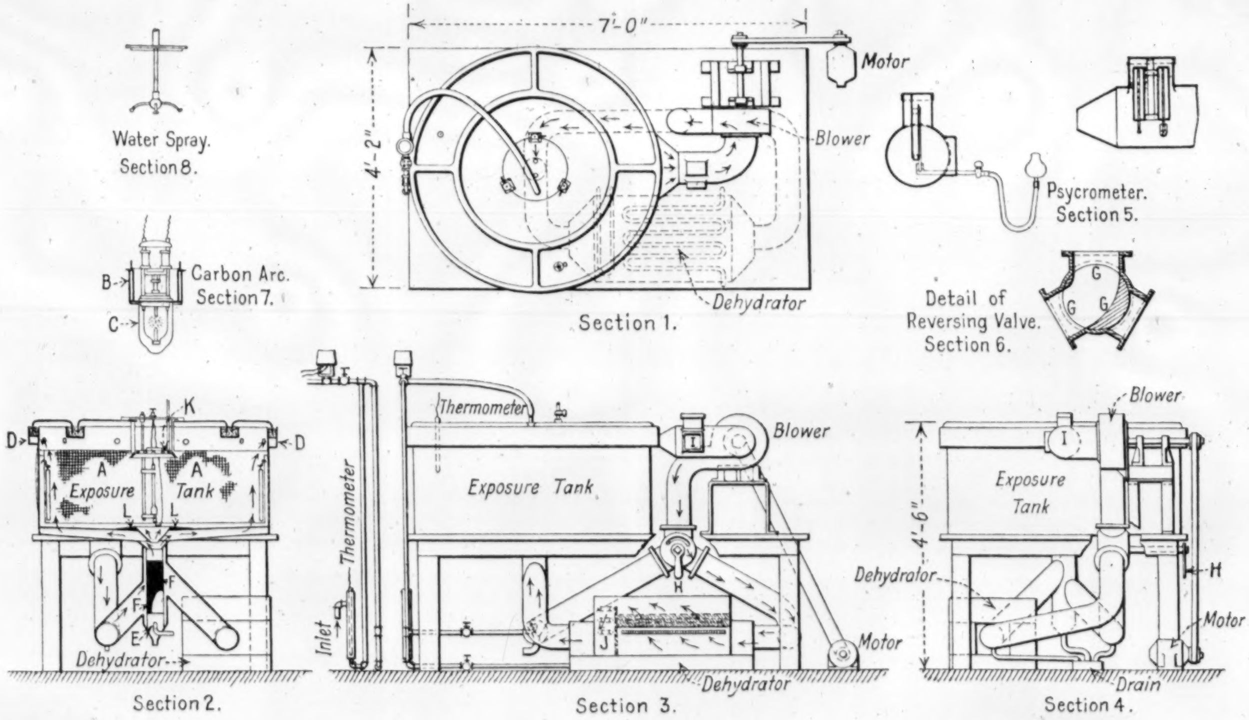
Water Spray.
Section 8.
B
C
Carbon Arc.
Section 7.
7'-0"
4'-2"
Motor
Blower
Dehydrator
Section 1.
Psycrometer.
Section 5.
Detail of
Reversing Valve.
Section 6.
G
G
G
D
D
K
A
A
Exposure
Tank
L
L
F
F
E
Dehydrator
Section 2.
Thermometer
Inlet
Thermometer
Exposure Tank
I
Blower
H
J
Motor
Dehydrator
Section 3.
Blower
I
Exposure
Tank
4'-6"
Dehydrator
H
Motor
Drain
Section 4.

FIG. 1.—Plan Drawing of Accelerated Weathering Equipment.

The top is made up of five removable covers: four small ones along the circumference for inserting the test panels, and a large one in the center for removal in case of repairs (Section 1, Fig. 1). These covers are mounted in sand seals to make the tank fairly gas tight at atmospheric pressures. The center of the large cover has a removable plate whereon are mounted, as required, the light sources and water sprays. It is a flat piece of metal machined at the joint and made sufficiently gas tight by means of clamps and an asbestos gasket.

The test panels are hung on a piece of ordinary galvanized $\frac{1}{4}$-in. mesh screening fixed to the wall of the exposure tank about 1 in. from the metal (*A*, Section 2, Fig. 1).[1]

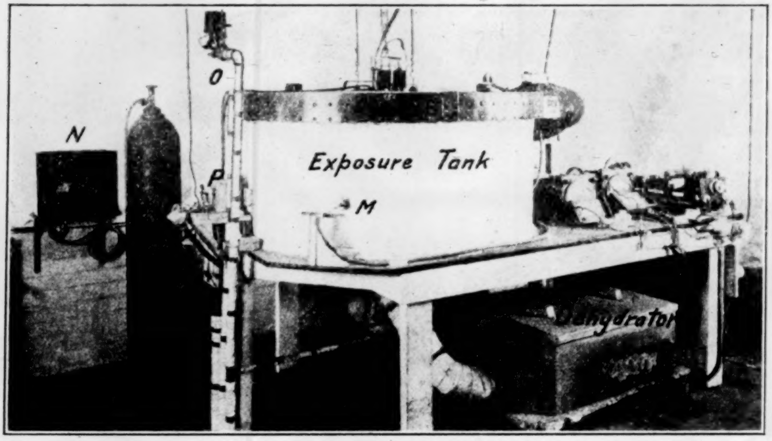

FIG. 2.—Photograph of Apparatus with Carbon Arc in Place.

In order to make the temperature control inside the tank independent of room temperature the apparatus is insulated. The exposure chamber has a removable false bottom in order to facilitate the control of the air circulation, as explained later.

Light Source.—Inasmuch as no destructive light source entirely representative of sunlight has as yet been made available for use in an accelerated weathering system, this apparatus has been designed for applying either of the two light sources now most generally used by the trade for such work. They are the quartz mercury arc[2] and the flaming arc with treated electrodes.[3] As indicated (Section 2,

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[1] This system of supporting the test surfaces is used in the accelerated weathering equipment of Bird and Son, Inc., East Walpole, Mass.

[2] Cooper-Hewitt vertical "Uviarc."

[3] Atlas Electric Devices Co. "Fade-O-Meter" arc.

Fig. 1), the mercury arc is attached to the center cover plate and lighted by lifting out the plate and lamp and tilting. Care must be exercised not to tilt the lamp beyond the horizontal in lighting, otherwise cathode arcing might injure the burner. A fixed automatic tilting device (*M*, Fig. 2) has been in use in the laboratory for some time and can be readily inserted, but it complicates the apparatus as a whole.

The carbon arc requires outside cooling, which is provided for by the water jacket on the supporting collar (*B*, Section 7, Fig. 1). The dash-pot control is inside the supporting collar, and the resistances are mounted at some more distant point. The glass globe surrounding the arc (*C*, Section 7, Fig. 1) may be fastened at the top with a removable gasketed ring so as to make the space within the globe a fairly gas-tight unit.

Air Conditioning.—The whole system is a closed unit working at atmospheric pressure. Intermediate steps in air conditioning are avoided since they complicate the apparatus and serve no real purpose in the test and only air of 100 per cent relative humidity or less than 5 per cent relative humidity is used. The air flow for high humidity conditions is indicated by the arrows in Sections 1 and 2, Fig. 1. The air is drawn out at the circumference of the tank through channel *D*, Section 2, Fig. 1. On the return, a small portion of it is forced directly upward to cool the mercury arc (this is shut off when the carbon arc is used) while the larger amount passes below the false bottom and enters the tank through perforations at the bases of the panels and sweeps over the test surfaces. A finely atomized spray of water introduced at *E* constitutes the humidifier. To prevent the carrying over of large particles of water and also to insure complete mixing of spray and air, the spray is enclosed in a wire screen cylinder and the air forced through the screen *F*. To insure complete saturation of the air, the temperature of the spray water should be a few degrees higher than the operating tank temperature. This can readily be accomplished by passing the water supply line through a steam jacket.

Low humidity conditions are obtained by means of silica gel, which eliminates air conditioning by refrigeration.[1] The ease with which this material may be reactivated by heating to approximately 250° F. in a current of air makes it a very convenient agent. Because of the exothermic nature of the water-adsorption effect of the silica gel, some cooling is desirable during the dehydration process. According to calculations based upon data given in the literature,[1] 1 lb. of silica gel at 86° F., activated until it has an initial water content of 7 per cent by weight, will dehydrate saturated air at 86° F. to 1 per cent relative humidity at the rate of approximately 0.8 cu. ft. per minute for a total time of 2 hours. In other words, at 86° F. 1 lb. of silica gel will dehydrate 96 cu. ft. of saturated air with 99 per cent efficiency. Because the apparatus represented in Fig. 1 is designed as a closed system using only a fixed volume of approximately 25 cu. ft. of air, the amount of gel required is comparatively small. Experimentally, about 15 lb. of silica gel has been sufficient to take care of the dehydrating requirements of the apparatus for about one week's run covering 48 hours of actual usage at less than 5 per cent relative humidity. The arrows in Section 3, Fig. 1, indicate the course followed by the air during a low-humidity exposure period. The dehydrator consists of a box with a middle shelf of wire screening upon which the silica gel is placed to a depth of about 4 in., interspersed with sections of loose screening to prevent packing. Below the shelf is a resistance unit for heating the gel and above, within the gel, a cold water coil for cooling. If high-pressure steam is available, the same coil can be used for heating and cooling and the resistance unit dispensed with.

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[1] Silica gel was first tried for this purpose at the suggestion of F. W. Faragher of the Mellon Institute of Industrial Research and a dehydrator of this type has also been incorporated in accelerated weathering equipment recently designed at that laboratory.

To change from high humidity conditions to low, or the reverse, it is necessary only to change the setting of the uni-control valve shonw in Section 6, Fig. 1 and *H*, Sections 3 and 4, Fig. 1. This valve is an aluminum casting having a movable center core G with machined sides so as to be air tight.

Wet and dry bulb thermometers have been inserted in the recirculating system at *I*, Sections 2, 3 and 4, Fig. 1. The wick of the wet bulb is wetted by means of an outside leveling bottle control as detailed in Section 5, Fig. 1.

Temperature Control.—The temperature during the light exposures has been established, rather arbitrarily for the present, as 140° F. On September 1, 1925, a clear day with a temperature reading in the direct sunlight at noon of 82° F., some surface temperature measurements on a red, a white and a black surface read 134.5, 115 and 142° F., respectively. In *Circular No. 296* of the U. S. Bureau of Standards, p. 10, it is stated that the measurements on the roof of the National Museum, Washington, D. C., showed that under direct sunlight the roof attains a temperature 50° F. higher than that of surrounding air.

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[1] E. B. Miller, "Adsorption of Silica Gel—Theory and Application," Am. Inst. Chemical Engrs., Vol. 13, Part I, p. 379 (1920).

The heat for maintaining the exposure temperatures is derived mainly from the light source. In the case of the carbon arc so much heat is dissipated that auxiliary water cooling is necessary, as previously mentioned. When running under dry conditions some reheating of the air is required after passing through the cooled silica gel. This is accomplished by the steam coils at *J*, Section 3, Fig. 1. In an insulated system, a close control on the tank temperature is attained by adjusting the blower speed.

Spraying Panels with Water.—In this laboratory, actual washing and water spray erosion of the test surfaces is carried on in a separate unit, because the tying up of the light exposure unit for such purposes is not considered an economy, but it is quite feasible to do this in the light exposure tank. An ordinary small lawn spray is attached to the interchangeable center plate as indicated in Section 8, Fig. 1. Warming the spray water to about 90° F. greatly increases the destructive action of this exposure without upsetting its simulation to actual weathering conditions. Of course, the air recirculating system is not used during the water-exposure period.

Wetting Test Surfaces While Exposed to Light.—In some humid southern climates, at certain periods of the year, paint surfaces are wetted four or five times a day by rains of very short duration followed immediately by strong sunlight. Besides, it often happens that wet surfaces are allowed to dry under direct sunlight. The authors have previously found[1] that light playing upon a water-soaked surface is one of the most destructive combinations in the weathering system. With the simulation of such conditions in mind, a ring of water atomizers has been attached to the center plate, as indicated at *K* in Section 2, Fig. 1, for use with the mercury arc. By means of a time clock and solonoid valve arrangement (*N* and *O*, Fig. 2), it is possible to wet the test surfaces periodically while exposed to light. As a precaution against water splattering the quartz, a protective shield is installed above the light and a wire screen collar below as at *L*, Section 2, Fig. 1.

Temperature Change Exposures (Refrigeration).—From the viewpoint of thermal efficiency, it certainly would be impractical and wasteful to attempt to cool all or any part of the light and water exposure unit even to a moderately low temperature. This important part of the accelerated system must still be carried out in a separate low-temperature cabinet.

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[1] H. A. Nelson and F. C. Schmutz, "Further Study of Accelerated Weathering: Effect of Variations in Exposure Cycle Combinations on Common Types of Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 920 (1924).

The Use of Oxygen-Enriched Atmospheres.—Until recently the common types of deterioration such as paint chalking and varnish and lacquer cracking, were all reproducible in the accelerated system with the exception of the fine hair-line checking so often found on weathered paint surfaces. A study of this last type of failure easily leads to the conclusion that it is induced through excessive hardening or contraction of the paint at the surface as compared with the interior portions of the film. With a view toward emphasizing the conditions under which surface oxidation might be accelerated in the same proportion as the other deteriorating factors are intensified, the oxygen content of the air in the light-exposure tank was increased to approximately 30 per cent by volume. Other exposure conditions were kept the same. The checking type of deterioration of ordinary outdoor paints was thus more readily reproduced. Figure 3 shows some photomicrographs of representative failures on outdoor paints as obtained by the accelerated weathering system now in use.

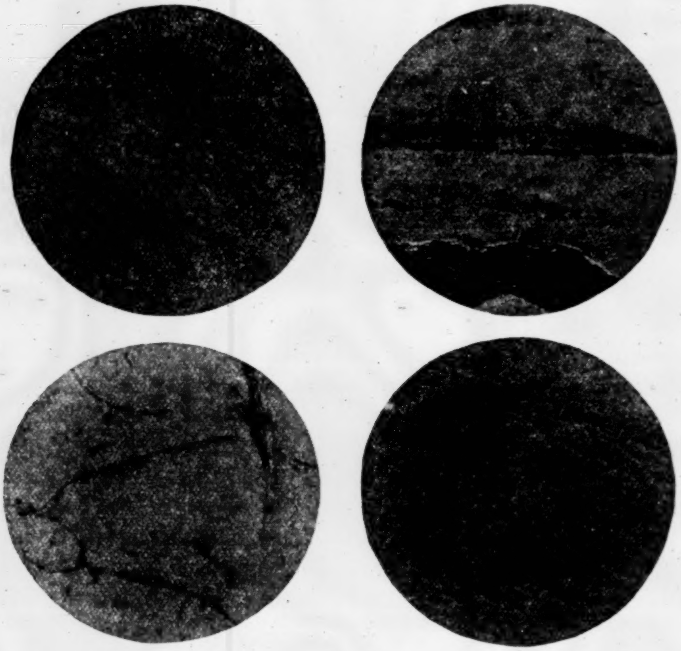

FIG. 3.—Representative Failures on Exterior Paints as Produced in the Accelerated Weathering System. (× 10.)

In addition to developing paint checking, the use of oxygen has also speeded up the deterioration rate considerably. Whereas previous to the use of this gas, our tentative standard cycle, which will be explained later, seemed to bear a deterioration rate ratio to outdoor exposures of about 7 to 1, since the enrichment this ratio seems to have become about 15 to 1. These figures, obtained when using the mercury arc as a light source, are only tentative in that it will require more experimental observation to fix the ratio more exactly.

It is not known whether 30 per cent represents the most desirable oxygen concentration. It is possible that similar results can be obtained with less enrichment. It has recently been suggested[1] that ozonized air might be used in place of oxygen. Since work along this line has just been started, no definite results can be reported, but the indications are rather favorable. From time to time during the very first few hours of the life of the mercury arc, the presence of ozone is indicated by odor. Tests for ozone by an approved method[2] have proved that the quantities of ozone formed due to ionization of air by the light are so small and persist for so short a time that it probably would have a negligible effect upon the deterioration process. The amount found during the first ten hours that a new burner was in use was 0.00255 per cent by volume (standard conditions). This volume dropped to 0.00045 per cent after 16 hours and was less than a determinable quantity after 17 hours.

DERIVATION OF AN EXPOSURE CYCLE

The next step in the application of the apparatus is the development of a practical system of manipulation or exposure cycle for which the following scheme is presented for consideration.

Investigation of local weather-bureau data for Palmerton and the vicinity for the year between December 1, 1924 and December 1, 1925, reveals the following significant figures:

1. There occurred 168 temperature cycles, or an averaged sequence of a maximum and minimum temperature every two to three days.
2. There were 2429 actual hours of sunlight and 630 hours of rain (hours of snow counted as rain).
3. The average absolute humidity was 3.317 grains per cu. ft. of air; 216 days were below this average and 149 days above. The average relative humidity was 73.5 per cent.

From a routine point of view the week lends itself most readily as a basic unit of time for constructing the cycle. Experimentally it has been found that, for the panels used (selected white pine, 12 by

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[1] By Mr. F. W. Hopkins, The Murphy Varnish Co., Newark, N. J.

[2] United States Ozone Co., Scottdale, Pa., *Technical Publication No. 14*.

6 by $\frac{3}{4}$ in., or No. 22 gage metal, 12 by 6 in.), about 2 hours should be allowed to bring the test panels from a temperature of 140° F., attained under the light, to a temperature of 0° F. Thus it becomes a physical impossibility to include the 168 temperature changes called for by the climatological data, all in a weekly cycle. One temperature change per day has been accepted as a compromise representing, at least, the change from the maximum day temperature to the minimum night temperature.

Except for the short time allowed for changing panels and making observations, the remainder of the week is divided between light and water exposures; 3.45 hours of the former to 1 hour of the latter, as determined by the figures given above for the hours of sunlight and rain during the year.

In dividing the total time for light exposure between humid and dry periods, the following salient points must be taken into consideration:

1. The high relative humidity (100 per cent) used in the accelerated system is approximated in this vicinity. Hence, the deteriorating action of the high humidity conditions in both cases should be about the same.
2. The low relative humidity (less than 5 per cent) is never attained outdoors hereabouts. In this case, then, this deteriorating action in the tank must be much more severe. (In a drier climate this discrepancy will not be so great.)
3. Some correction must be applied to equalize this differential in effectiveness. This can be done by adjusting the relative times of exposure allotted to each condition.
4. The difference between the 100 per cent relative humidity, used in the accelerated system, and the average outdoor relative humidity is 26.5 per cent. The difference between the low relative humidity and the outdoor average is 68.5 per cent. These figures, applied as an inverse ratio, 1:2.58, should approximate the equalization necessary to make both exposures equally effective.
5. According to the climatological data, the ratio of humid days to dry days is 1:1.45. From the reasoning in (4) the time thus allotted to dry conditions should be reduced to 38.8 of its original value or 0.56.
6. The total light exposure time should accordingly be divided approximately into 1 part high humidity to 0.56 part low humidity.

The total actual running time per week allowed at present is 162 hours. In view of the previous calculations, this running time is divided up as follows: 12 hours refrigeration (2 hours per day exclusive of Sunday), 31 hours of water spray exposure at 90° F., and 119 hours of light exposure at 140° F. Of the last, about 76 hours is at 100 per cent relative humidity and 43 hours at less than 5 per cent relative humidity.

The sequence in which these weathering factors are to be arranged in the cycle must now be arrived at. From previous investigations,[1] it has been found that the most effective sequences are: (1) a water-spray exposure followed by a light exposure, (2) water to a refrigeration change and, (3) light to refrigeration; while the less effective sequences are: (4) light to water, (5) refrigeration to water, and (6) refrigeration to light. Thus, it is desirable to have sequences (1), (2) and (3) in predominance. The actual hours that each weathering factor can be used per day is limited by the working conditions. As previously explained, one refrigeration (temperature change exposure) per day, exclusive of Sunday, is fixed. Also, because of the length of the light-exposure periods, these will preferably be taken care of during the night. The rest of the cycle can now be readily filled in.

Table I.—Tentative Cycle Approximating Weather Conditions.

Day	Weathering Factor	Duration
Monday	Refrigeration 0° F.	2 hours
	Water 90° F.	3 hours
	Light 140° F.	18 hours (16 hours wet, 2 hours dry)
Tuesday	Water	3 hours
	Refrigeration	2 hours
	Light	18 hours (16 hours wet, 2 hours dry)
Wednesday	Refrigeration	2 hours
	Light	3 hours (wet)
	Water	16 hours
Thursday	Refrigeration	2 hours
	Water	3 hours
	Light	20 hours (wet)
Friday	Refrigeration	2 hours
	Water	3 hours
	Light	18 hours (wet)
Saturday	Refrigeration	2 hours
	Water	3 hours
	Light	15 hours (dry)
Sunday to Monday	Light	27 hours (24 hours dry, 3 hours wet)

A tentative cycle thus developed to approximate the weathering conditions in the vicinity of Palmerton for the year between December 1, 1924 and December 1, 1925, may be as summarized in Table I.

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[1] H. A. Nelson and F. C. Schmutz, "Further Study of Accelerated Weathering: Effect of Variations in Exposure Cycle Combinations on Common Types of Varnishes," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 920 (1924).

The same line of reasoning can be followed in developing a cycle for any locality, but it would be desirable to average the climatological data for more than one year. The periodic wetting while under a high humidity light exposure, previously referred to, is not applicable in the above cycle, but might be in a cycle designed to simulate conditions in Florida, for example.

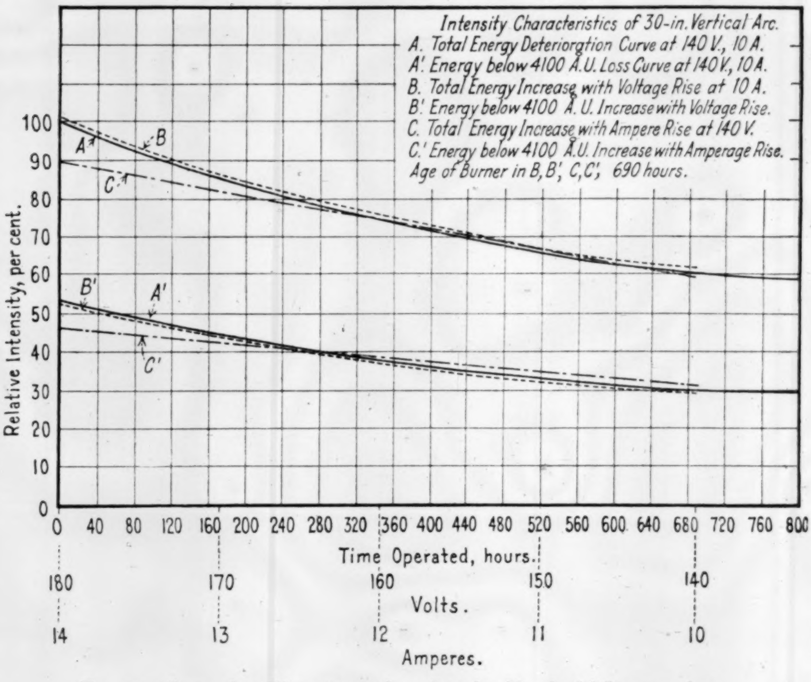

FIG. 4.—Intensity Characteristics of 30-in. Vertical Mercury Arc.

CHANGES IN THE UVIARC WITH USE

The gradual dropping off in intensity of the mercury arc burners due to clouding of the quartz is a practical weakness in their use for accelerated testing purposes. O. R. Flynn[1] recommends a gradual increase in the electrical energy input to compensate for the intensity loss.

By means of a Hilger type F 110 linear thermopile (P, Fig. 2) and a sensitive D'Arsonval type of galvanometer, an accurate record has been kept of the decrease in intensity during the first 800 hours usage of a 30-in vertical arc running at 140 volts and 10 amperes. The various portions of the spectrum were studied by means of corning glass filters. The curves *A* and *A′* in Fig. 4 represent the falling off in total energy and also in energy below 4100 Ångström units, respectively. The region below 4100 Ångström units was plotted because there is evidence that energy from this part of the spectrum is mainly responsible for the rapid acceleration of the reactions that contribute to the breaking down of organic surface finishes.

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[1] O. R. Flynn, "How the Mercury Arc Was Made to Imitate Sunlight," *American Dyestuffs Reporter*, Vol. XII, pp. 837–843, November 19, 1923.

It should be noted that in both cases the slopes of the depreciation curves become negligible at about 800 hours. The graph indicates that the intensity of a depreciated 30-in. burner can be made to equal a new burner quite satisfactorily by increasing the voltage in particular. The voltage in the arc can be increased by decreasing the external resistance in the circuit and operating the burner with less cooling. The amperage can be raised by increasing the external resistance and also the cooling.

We wish to express our appreciation to Mr. G. F. A. Stutz and other members of our Research Organization for their cooperation and assistance in this work.

DISCUSSION

A Member. A MEMBER.—Would it not be possible to determine the relative effect of the different wave lengths and to base subsequent research upon the effective radiations?

Mr. Nelson. MR. H. A. NELSON.—From the work that has been done along this line we know, with a fair degree of certainty, that the really effective radiations are those below about 4500 to 4100 Ångström units. The effectiveness of the radiations tends to increase as the radiations decrease in wave length, but whether the increase is inversely proportional throughout we do not know. There is evidence that it reaches a maximum between 3500 and 2500 Ångström units.

Mr. Busby. MR. H. S. BUSBY.[1]—I should like to say, in support of Mr. Nelson's statement, that a great deal of work has been done on those very regions to determine, for a wide range of coloring matters, what the effect is of the ultra violet light. The *Textile Colorist* of March, 1923, discusses it in considerable detail, and I think it will be found that there will be no difficulty in assigning proper weights to different regions of the spectrum.

Mr. Wertz. MR. F. A. WERTZ.[2]—Has Mr. Nelson done anything with the iron arc, which I believe is supposed to be the richest of all in ultraviolet light? I should also like to ask whether finding that after 17 hours no more ozone is produced, is consistent with what is usually considered the cause of the production of ozone? Is it not the ultra violet rays that convert the oxygen to ozone? My limited experience with quartz mercury lamps is that the characteristic odor of ozone persists, no matter how long the lamp is run.

Mr. Nelson. MR. NELSON.—We were not able to detect ozone in the apparatus by the approved methods used in testing for ozone, after the burner had been used 17 to 20 hours (not a continuous run). The ionizing radiations for oxygen in air are down around 1800 Ångström units, so it is quite possible that the quartz clouds so rapidly that these radiations soon become too weak to be effective even at the surface of the quartz.

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[1] Director, A. French Textile School, Georgia School of Technology, Atlanta, Ga.

[2] Production Manager, Varcraft Works, Inc., Pottstown, Pa.

A STUDY OF CERTAIN FACTORS AFFECTING THE STABILITY OF ASPHALT PAVING MIXTURES

By Prévost Hubbard[1] and F. C. Field[2]

Synopsis

In 1925, the authors presented a paper describing a laboratory test which they had devised for determining the relative stability or resistance to displacement of paving mixtures of the sheet asphalt type. By means of this test it has been possible to study various factors affecting the stability of paving mixtures and the present paper presents the results of such study in connection with the following:

1. Effect of variations in percentage of mineral filler and asphalt cement with a given sand aggregate.
2. Effect of variation in penetration, ductility and melting point of asphalt combined with different mineral aggregates.
3. Effect of variations in temperature of mixture when tested.

In addition a new method of determining voids in mineral aggregates is described and the relation of voidage to stability is discussed. From this investigation the following deductions have been drawn:

1. Closeness of packing or reduction of voids by compression of the mineral aggregate exerts a much greater stabilizing effect than the mere reduction of voids in the entire mixture. Specific gravity or density of the mixture itself is therefore not always a direct indication of either degree of compression or stability.
2. Asphalt films surrounding the particles of compressed mineral aggregates may be thinner than the air or liquid films which exist in the usual methods of determining voids. Therefore any attempt to proportion asphalt in a mixture with the idea of completely filling the voids as determined in the mineral aggregate may result in the use of too much asphalt with a marked reduction in stability.
3. The greatest stability for a given mineral aggregate has not been obtained with that proportion of asphalt which would produce the lowest percentage of voids in the compressed mixture.
4. For maximum stability in paving practice the percentage of asphalt must be controlled within much narrower limits for high-filler low-void aggregates than for relativity low-filler high-void aggregates.
5. Of the physical characteristics of asphalt usually determined, penetration at 77° F. appears to have the most direct and definite effect upon stability of the mixture at all temperatures and a general increase in stability, produced with a given mineral aggregate, accompanies decrease in penetration of the asphalt.
6. Next to closeness of packing of the mineral aggregate, percentage of filler appears to be the greatest stabilizing factor and provided the proper pro-

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[1] Chemical Engineer, The Asphalt Association, New York City.

[2] Chemist, The Asphalt Association, New York City.

portion of asphalt is used, increased stability is invariably produced by increase in filler at least to the extent that such addition decreases voids in the mineral aggregate. If, however, the proper proportion of asphalt is not used, an increase in filler may result in decrease in stability.

7. The stability of a given compressed mixture appears to be directly proportional to its temperature, but the proportion varies for different mixtures so that of two mixtures which show the same stability at 140° F. one may be much harder and more likely to crack at low temperatures than the other.

At the 1925 annual meeting of the Society the authors presented a paper[1] describing a laboratory test for determining the relative stability, or resistance to displacement, of paving mixtures of the sheet asphalt type. This test consists in forming a cylindrical briquet from the hot paving mixture, allowing it to cool and age for a short period, then warming it to 140° F., placing it in a testing mold and measuring the maximum load required to force the mixture through a circular orifice of given diameter. This load is recorded as the stability value of the mixture.

Asphalt paving mixtures of this type, which are composed of sand, mineral dust and asphalt cement, are plastic bodies meeting Bingham's[2] definition of a plastic solid which is as follows: "A plastic solid is made up of particles which touch each other at certain points. The spaces between these particles may be empty or may be filled with gas, liquid, or amorphous solid." Bingham defines plasticity as "a property of solids by virtue of which they hold their shape permanently under the action of small shearing stresses but they are readily deformed, worked or molded under somewhat larger stresses." He further calls attention to the fact that plasticity is a complex property made up of two independent factors, internal friction and mobility, and that in plastic flow it is generally understood that a definite shearing force is required before any deformation takes place.

In the test devised by the authors measurement is made of the initial resistance of the mixture to shear, or of its initial internal friction, and not of its mobility or plastic flow. Other things being equal, it is evident that this initial internal friction will depend to a great extent upon the closeness of packing of the mineral particles. In other words, degree of compression or density is a most important factor bearing upon the stability of any given mixture, and this was clearly indicated in a series of tests by the authors on briquets of the same mixture formed under direct pressures of 1000, 2000 and 3000 lb. per sq. in., where it was found that the stability value of the mixture varied directly with the forming load. Of course such relation would not continue to hold indefinitely with increase in pressure, as a point would finally be reached where no greater density could be secured without crushing the individual grains of the mineral aggregate. Such a point may be termed maximum practical density.

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[1] Prévost Hubbard and F. C. Field, "A Practical Method for Determining the Relative Stability of Fine-Aggregate Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 335 (1925).

[2] "Fluidity and Plasticity," by Eugene C. Bingham, McGraw-Hill Book Co.

Direct compression under 3000 lb. per sq. in. had originally been selected as a basis for comparing stability values, but it was later noted that under this load many mixtures did not develop the density that they might reasonably be expected to possess and this fact has led to a modification of the original method of preparing specimens. This modified method, which is briefly described in the following paragraph has, in general, raised the stability value of all mixtures tested, but where checks have been run the trend of new results is practically the same as for the old results and the differences are for the most part purely relative.

The following directions cover the new method of preparing test specimens in batches of three, each batch weighing approximately 350 g.

> "Weigh out the dried sand and filler to 0.1 g. Heat this aggregate in the tared mixing dish to 350° F., place on balance and weigh in hot asphalt at 350° F. to 0.1 g. Thoroughly mix the contents of the dish for 5 minutes with a 1-in. putty knife, then place approximately 100 g. in each of three forming molds, numbered 1, 2 and 3, preheated to 250° F. Place the molds with base plates on a hot plate and bring the contents of No. 1 mold to 275° F. as determined by a thermometer which is used for stirring the mixture. Remove mold No. 1 from the hot plate and tamp the contents for ½ minute with a ¾-in. diameter round iron rod 8 in. long. Then insert the plunger and place the mold in the empty water bather on the base of the compression machine. Apply pressure to the top of the plunger until a load of 3000 lb. per sq. in. is reached. Fill the bath with cold water to a depth of 3 in. and allow the briquet to cool under pressure for 5 minutes. Then release the pressure, remove the mold from the bath and force out the briquet. While the first briquet is under pressure bring the contents of No. 2 mold to 275° F. Tamp, compress and cool the mixture as in the case of No. 1. Repeat the operation with No. 3 mold. Keep the hot plate so regulated that until the mixtures are to be brought to the temperature for tamping they are maintained at about 170 to 180° F."

The compressed briquets should measure approximately 1 in. in height. After removal from the mold they are allowed to age in air over night and then placed in a hot water bath accurately maintained at the temperature of test (usually 140° F.) for not less than one hour before testing.

The new method of forming specimens has improved the accuracy of the test but occasional erratic results on briquets of the same mixture and of practically the same density have led the authors to believe that lack of uniform density throughout the individual test specimen may sometimes occur and that the method may eventually be further improved so as to insure maximum practical density throughout the entire briquet.

In the paper last year the authors showed by diagrams that the addition of increasing amounts of mineral filler to a given sand in general increased the stability of certain asphalt paving mixtures and that different fillers developed different stability values in the mix when present in the same percentage by weight. Thus, a mixture containing 10 per cent of hydrated lime showed a stability value about equal to one containing 20 per cent of limestone dust and about one and one-half times as great as a mixture containing 10 per cent of limestone dust.

TABLE I.—PHYSICAL CHARACTERISTICS OF SAND AND FILLER.

MECHANICAL ANALYSIS:	CRANE SAND	LIMESTONE FILLER
Passing 10 mesh, retained on 20 mesh	2.5 per cent	...
Passing 20 mesh, retained on 30 mesh	7.4 per cent	...
Passing 30 mesh, retained on 40 mesh	11.5 per cent	...
Passing 40 mesh, retained on 50 mesh	20.2 per cent	0.2 per cent
Passing 50 mesh, retained on 80 mesh	36.2 per cent	0.5 per cent
Passing 80 mesh, retained on 100 mesh	12.0 per cent	1.9 per cent
Passing 100 mesh, retained on 200 mesh	9.4 per cent	25.5 per cent
Passing 200 mesh, retained on 250 mesh	0.8 per cent	5.3 per cent
Passing 250 mesh, retained on 300 mesh	...	1.6 per cent
Passing 300 mesh, retained on 350 mesh	...	9.4 per cent
Passing 350 mesh	...	55.6 per cent
Total	100.0 per cent	100.0 per cent
Specific Gravity	2.64	2.69
Voids, per cent	38.8	29.0

In a later paper[1] the authors showed that when the percentage of asphalt in the mix is kept constant and mineral filler is added to a given sand in increasing amounts, a point may be reached where further addition of filler begins to lower the stability. From a series of mixtures containing varying percentages of the same constituents it was further shown, within certain limits, that among compressed mixtures containing the same percentage of voids, mixtures which showed the lowest percentage of asphalt developed the greatest stability. Typical curves were also shown illustrating the following. With a given mineral aggregate maximum stability is secured in mixtures carrying a rather limited range of percentage of asphalt. Increase in stability is to some extent controlled by increase in hardness of asphalt.

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[1] Prévost Hubbard and F. C. Field, "Researches on Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Municipal Improvements, 1925.

As the beginning of a systematic study of the many factors bearing directly upon the stability of asphalt paving mixtures, it was decided to concentrate upon the effect upon stability of variations in characteristics and percentage of the asphalt cement. The results presented in this paper deal primarily with this phase of the investigation although it will be seen that most of the other factors already mentioned must be considered at the same time. All of the mixtures hereafter described contained the same sand and limestone filler, possessing characteristics as shown in Table I. The sand represents a product widely used in sheet asphalt construction in New York City and vicinity, while the limestone dust, although containing somewhat less material passing the 200-mesh sieve than desirable, is widely used in the same locality and was obtained from the plant of a large paving contractor.

TABLE II.—PHYSICAL CHARACTERISTICS OF ASPHALT CEMENT.

Sample	Materials	Penetration at 77° F.	Specific Gravity	Melting Point, deg. Fahr.	Ductility, cm.		
					32° F.	77° F.	115° F.
No. 1...	Brick Filler	28	1.034	165	1	9.3	20
No. 2...	Waterproofing Asphalt	35	1.04	152	4	33.5	107
No. 3...	Paving Asphalt, 31 to 40 Penetration	37	1.044	135	1.5	103	130
No. 4...	Paving Asphalt, 41 to 50 Penetration	48	1.041	133	1.75	101	150+
No. 5...	Paving Asphalt, 61 to 70 Penetration	66	1.034	125	4.5	106	150+
No. 7...	Paving Asphalt, Fluxed	106	1.035	113	9.0	80	110

The asphalts used in these tests were all produced from crude Mexican petroleum and possessed the physical characteristics shown in Table II. Samples Nos. 3, 4 and 5 were regular steam-refined paving grades. Sample No. 7 was an asphalt cement produced by fluxing No. 5 with a heavy Mexican flux. Samples Nos. 1 and 2 were regular commercial grades of blown asphalt which were included in the tests for the purpose of ascertaining if low ductility of the bitumen had any direct influence upon the stability of paving mixtures in which they were incorporated.

In studying the effect of these asphalts upon the stability of paving mixtures it was decided to use three mineral aggregates composed of sand and 10, 20 and 30 per cent of limestone dust respectively.

Previous work had shown that stability was to a considerable extent dependent upon the percentage of asphalt in the mixture, so in order to select satisfactory mixtures for further comparison a series of tests was first run with mixtures composed of the three mineral aggregates and varying percentages of No. 4 asphalt. The results obtained are illustrated by the three curves in Fig. 1.

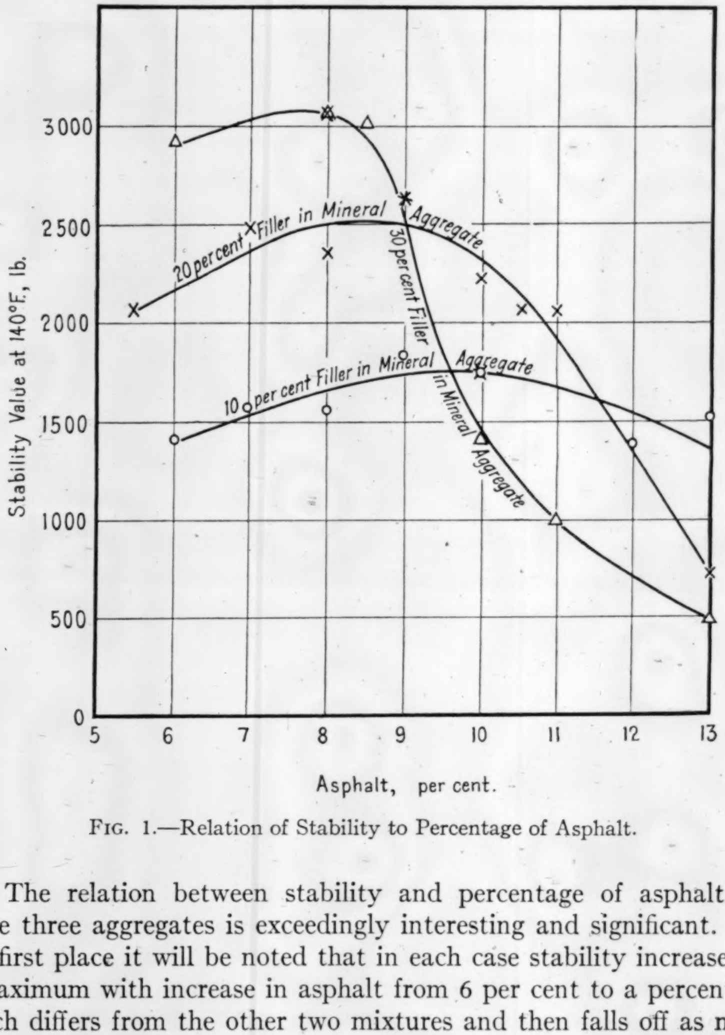

FIG. 1.—Relation of Stability to Percentage of Asphalt.

The relation between stability and percentage of asphalt for these three aggregates is exceedingly interesting and significant. In the first place it will be noted that in each case stability increases to a maximum with increase in asphalt from 6 per cent to a percentage which differs from the other two mixtures and then falls off as more asphalt is used. The stability represented at the crest of these curves is highest for the high filler mix and lowest for the low filler mix, but it is apparent that sensitiveness to slight changes in percentage of asphalt increases with increase in filler. In other words after maximum stability has been secured the high filler mixtures fall off much more rapidly with further increase in asphalt than do those containing less filler. The probable reasons for this will be discussed later as further data are presented. For the present it may be noted that as the amount of filler was increased from 10 to 30 per cent of the mineral aggregate, maximum stability was obtained by decreasing the percentage of asphalt in the mixture from 10 to 8 per cent. For this reason the mixtures shown in Table III were selected for further study.

At this point a slight digression may be helpful in analyzing the stability results so far presented and those which follow. A number of investigators have rather recently come to the conclusion that the percentage of voids in the mineral aggregate has a very direct bearing upon the amount of asphalt which can be successfully used in a given paving mixture. Various tests have been devised for determining voids in sheet asphalt aggregates, perhaps the most popular being methods in which the dry mineral aggregate is fed to refusal into a cone or cylinder of known capacity, the container being tapped or rapped upon a table to assist close packing. Voids in the packed aggregate are then calculated from the weight of the known volume, taking into account the specific gravity of the solid aggregate. By standardizing all details of procedure, reasonably satisfactory check results may be obtained, but the authors have felt that any method of packing a dry aggregate containing a high percentage of filler is likely to result in segregation to such an extent as to produce misleading results.

TABLE III.—COMPOSITION OF MIXTURES TESTED.

DETERMINATION	MIX NO. 1	MIX NO. 2	MIX NO. 3.
Passing 10 mesh, retained on 40 mesh...	17.0 per cent	15.0 per cent	14.0 per cent
Passing 40 mesh, retained on 80 mesh...	46.0 per cent	41.0 per cent	37.0 per cent
Passing 80 mesh, retained on 200 mesh..	20.0 per cent	21.0 per cent	21.0 per cent
Passing 200 mesh....................	7.0 per cent	14.0 per cent	20.0 per cent
Asphalt.............................	10.0 per cent	9.0 per cent	8.0 per cent
Total............................	100.0 per cent	100.0 per cent	100.0 per cent

As a means of obtaining some measure of voids in mineral aggregates as used in the stability test, a new method aimed to eliminate segregation in so far as possible was therefore devised. Preliminary tests were first run on dry sand containing no filler and on dry limestone dust, after which varying percentages of kerosine were mixed with each until a range in proportion of kerosine was established which would give the same calculated voids as found in the dry aggregates. The following is a brief description of the method which was thus developed:

Place 100 g. ± 0.01 g. of dry mineral aggregate in a porcelain saucer, weighing the sand and mineral filler separately in proper proportions where a combination of the two is to be tested. Add kerosine to the extent of 2.5 per cent of the weight of sand and 10 per cent of the weight of filler, and mix thoroughly. Then transfer the entire contents of the saucer to a cylindrical iron mold having an accurately determined diameter of approximately 5.08 cm. (2 in.). The forming mold employed in the stability test may be used for this purpose and should rest on a flat metal plate. Insert a close-fitting plunger in the mold and subject the mixture to a load of 3000 lb. per sq. in. for one minute. Release the load and measure the height of the compressed mixture in the mold to 0.01 cm. This may be done by subtracting the height of the plunger in the empty mold from its height when resting on the compressed mixture. Without reference to the kerosine present calculate voids in the mineral aggregate from the following formulas in which V = volume of compressed mixture in cubic centimeters, d = density of the compressed mineral aggregate, D = theoretical maximum density of the total mineral aggregate considered as a voidless solid, G and G' = the specific gravities of the sand and mineral filler, respectively, which must be predetermined by any of the well-known methods, and W and W' = the percentage weights of sand and mineral filler, respectively, which were present in the dry aggregate.

$$d = \frac{100}{V} \qquad (1)$$

$$D = \frac{100}{\frac{W}{G} + \frac{W'}{G'}} \qquad (2)$$

$$\text{Per Cent Voids} = \frac{100\,(D-d)}{D} \qquad (3)$$

Results obtained by this method on various combinations of the sand and limestone dust previously described are shown by the upper curve in Fig. 2 which is seen to be quite regular and shows a minimum percentage of voids in the aggregate containing 50 per cent each of sand and dust.

After it had been decided to tamp the asphalt mixtures for stability tests the same method of tamping was adopted for the kerosine mixtures used in void determinations with results shown by the second full curve which is seen to be almost parallel to the first curve but showing from 3 to 5 per cent lower voids for individual combinations. Relatively these two curves are the same, with corresponding high and low points so that either might be used as a general guide in selecting a practical combination of sand and dust which would approach the minimum percentage of voids as closely as possible.

It should be realized, however, that in any mineral aggregate, no matter how thoroughly compressed, the individual particles are not in absolute contact even at points but are separated by films—in the case of dry aggregates, air films, and in liquid mixtures, liquid films. While the authors felt that the kerosine method of determining voids represented approximately the same degree of close packing without segregation as any dry method, they felt that none of the methods might correctly show the degree of close packing of the mineral particles in an asphalt paving mixture as the films of asphalt might be greater or less than the films of air or kerosine. To obtain some information on this matter, they therefore adopted the following method of calculating voids in the mineral aggregate in asphalt mixtures, irrespective of whether these voids were completely or only partly filled with asphalt.

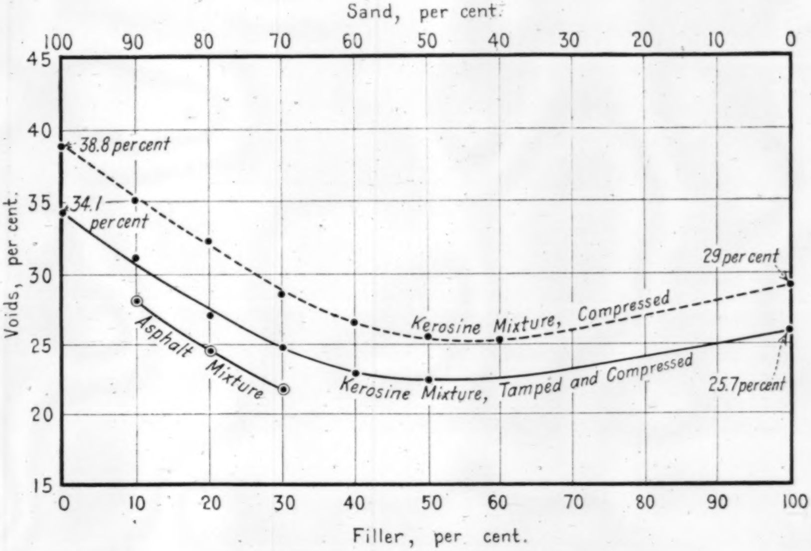

FIG. 2.—Effect of Filler on Percentage of Voids in Mineral Aggregate.

The specific gravity of the compressed asphalt briquet was first determined. From this determined weight of a unit volume, the weight of asphalt present was subtracted to obtain the weight or specific gravity of the same volume of mineral aggregate and the percentage voids in the mineral aggregate was then calculated by means of Eqs. 2 and 3. Results so obtained for the three asphalt mixtures that had been selected for further investigation are shown by the short curve in Fig. 2 where it is seen that the presence of asphalt in the amounts used further reduced the voids in the mineral aggregate. In other words, the mineral aggregate was more closely packed and the asphalt films separating the individual grains were thinner than either the films of air or kerosine. This is probably due to the relatively high viscosity and cementitiousness of the asphalt, but it is an important factor to be considered if the percentage of voids in the mineral aggregate is to set the maximum allowable amount of asphalt to be used in a paving mixture, which assumption has been made by a number of paving technologists.

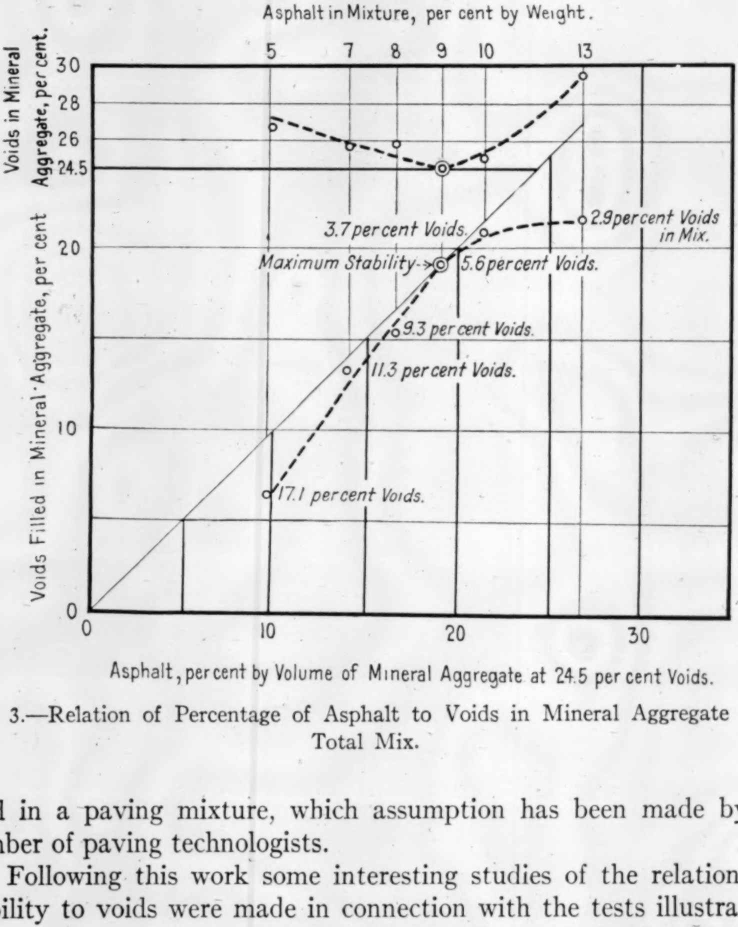

FIG. 3.—Relation of Percentage of Asphalt to Voids in Mineral Aggregate and Total Mix.

Following this work some interesting studies of the relation of stability to voids were made in connection with the tests illustrated in Fig. 1. For each compressed mixture the voids in the mixture itself were determined and the voids in the mineral aggregate, excluding the asphalt, were calculated to ascertain its closeness of packing. In each series it was found that maximum stability was developed where the mineral aggregate was most closely packed but that such mixtures did not show the lowest percentage of voids in the series. These relations are illustrated in Fig. 3 for the 20-per-cent filler series and are typical of those developed by the other two series.

If 24.5 per cent of voids in the asphalt-coated mineral aggregate, the lowest percentage obtained, is considered to represent maximum practical density it is seen from the upper curve that as the percentage of asphalt is increased from 5 per cent by weight, closeness of packing is gradually increased to a maximum at 9 per cent, after which it rapidly decreases with further addition of asphalt. Reference to the lower curve for which the asphalt has been calculated on a volume basis shows that further addition of asphalt decreases the percentage of voids in the total mixture but not in proportion to the volume increase in asphalt. It is believed that too little asphalt tends to prevent the closest possible packing of the mineral aggregate because of lack of mobility of the mixture during compression and that too much asphalt tends toward the same result by creating a pasty mixture which is apt to trap air. Thus, when more than 24.5 per cent by volume of the asphalt was used, or an amount which should have

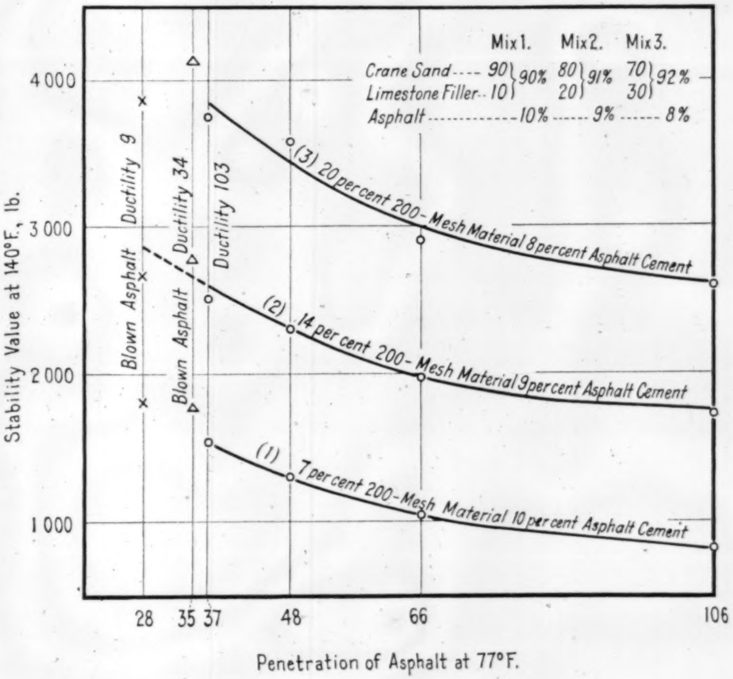

FIG. 4.—Relation of Stability to Hardness of Asphalt.

reduced the voids in the mixture to zero, nearly 3 per cent of voids was actually found.

The establishment of an apparently logical reason for the relatively high stabilities of mixtures Nos. 1, 2 and 3 justifies their selection for further comparative study. Figure 4 illustrates the effect upon their stability of variations in penetration of asphalt cement.

Here it is seen that there is a very similar and definite increase in stability with decrease in penetration (increase in hardness) of the steam-refined or fluxed asphalts of high ductility. Projections of these curves into the penetration range covered by the blown or oxidized asphalts of low ductility as in the case of mixture No. 2 indicates that the stability values obtained for the latter are only what might reasonably be expected from their lower penetration or greater hardness. In other words, no direct relation between stability and ductility can be established.

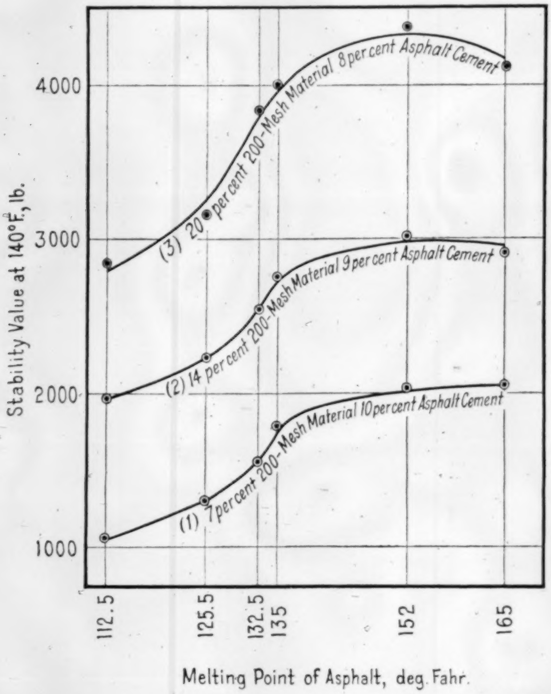

FIG. 5.—Relation of Stability to Melting Point of Asphalt.

Figure 5 shows that a fairly consistent relation between stability and melting point of the asphalt may be established but that it is not as regular or definite as the relation between stability and penetration.

In general, it is seen that stability increases most rapidly with increase in melting point up to the approximate temperature at which the test is made, after which further increase in melting point produces relatively less increase in stability. It should, of course, be realized that the so-called melting point is a misnomer, as asphalts have no true melting point. The test is therefore only an approximation of a temperature at which the asphalt reaches a certain rather indefinite degree of fluidity.

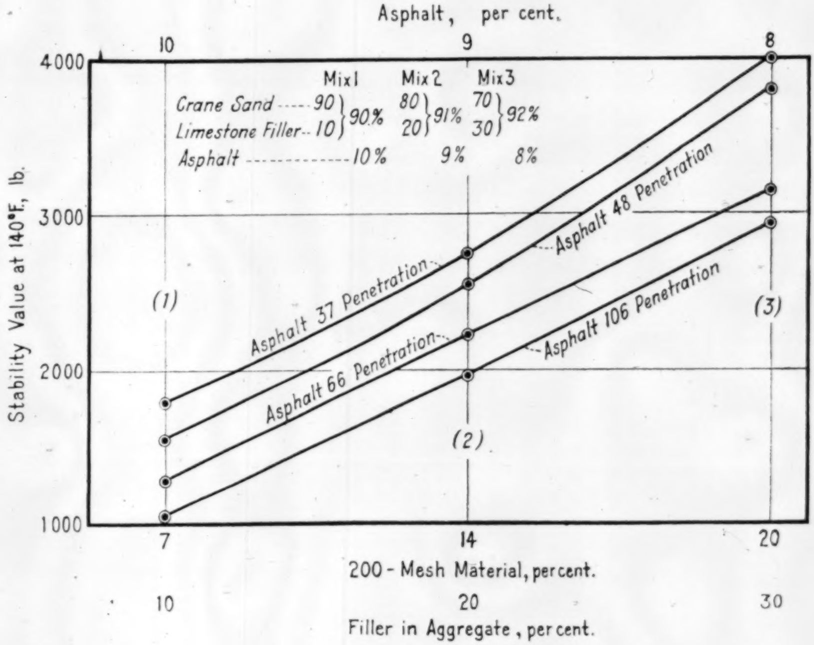

FIG. 6.—Relative Stability of Mixtures Made with Asphalts of Varying Hardness.

For any given penetration of asphalt the direct relation between stability and percentage of filler is more clearly shown in Fig. 6 than in the preceding figures. Here it is at once apparent that stability is more influenced by the percentage of filler than by even wide variation in characteristics of the asphalt cement. For instance, the stability of mixture No. 3 with the very soft asphalt of 106 penetration is higher than that for mixture No. 2 with a hard asphalt of 37 penetration. The possible significance of such relations will appear later.

That mere increase in filler will not necessarily increase stability unless the percentage of asphalt is adjusted accordingly is very clearly shown in Fig. 7 where the stability values given in Fig. 1 for the 8, 10 and 13-per-cent asphalt mixtures are plotted against the percentage of filler in the mineral aggregate. In this figure it will be noted that the stability values of the 10-per-cent filler mixtures are quite close together irrespective of the variation in asphalt between 8 and 13 per cent. In the 8-per-cent asphalt series stability increases consistently with increase in filler up to 30 per cent. In the 10-per-cent asphalt series stability increases with increase in filler to 20 per cent and then falls off at 30 per cent of filler, while in the 13-per-cent asphalt series stability is consistently reduced with addition of filler, which is exactly the reverse of the the 8-per-cent asphalt series. The probable reason for such variation in trend of results is now quite readily explainable from the previous discussion of voids or closeness of packing of the mineral aggregate.

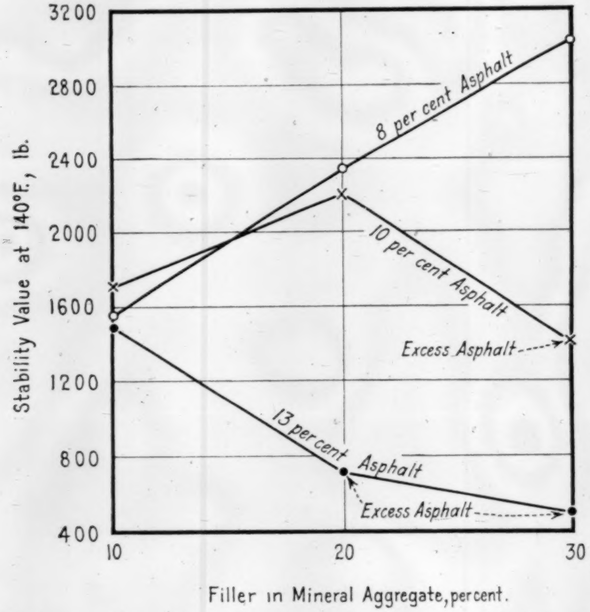

FIG. 7.—Effect on Stability of Relation of Percentage of Filler to Percentage of Asphalt.

Up to this point all of the stability tests which have been considered were made at a temperature of 140° F. It has been quite generally assumed that this temperature is about the maximum which the asphalt pavement reaches under ordinary conditions, but records of pavement temperature higher than 140° F. have been obtained by the Federal Bureau of Public Roads as far north as Washington, D. C., and it is quite probable that higher maximums occur for short periods in many parts of the United States. It was therefore considered advisable to obtain some idea of the effect of variation in temperature upon the stability of various mixtures. Accordingly a series of tests upon mixtures Nos. 1, 2 and 3 with the different asphalts was run at temperatures of 100, 140, 150 and 160° F. The relative effect of variation in temperature upon all three mixtures was found to be very similar and is illustrated by Fig. 8 covering mixture No. 3.

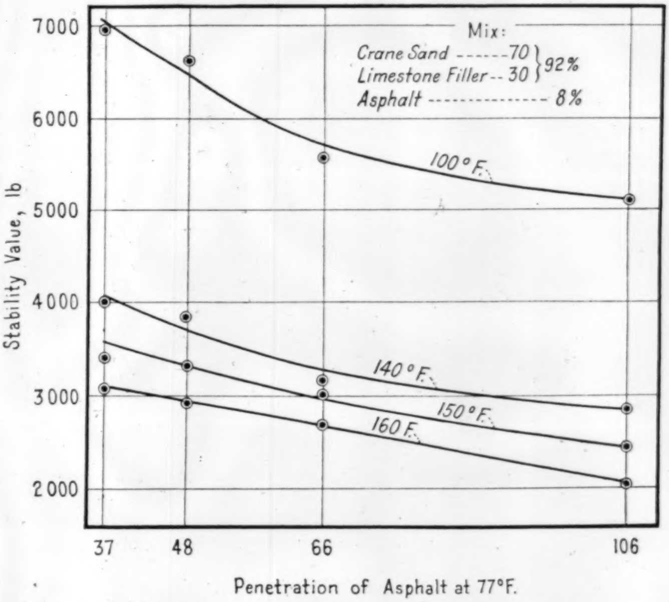

FIG. 8.—Relation of Stability to Temperature and Hardness of Asphalt in High Filler Mixes.

Here each curve represents a series of tests at a single temperature and shows the stability at such temperature as affected by the penetration of the asphalt cement used in the mixture. It will be noted that the same general type of curve occurs at all four temperatures and that stability is invariably increased with decrease in penetration of the asphalt, although as the temperature becomes less there is a tendency for the curves to become steeper. This, of course, means that at comparatively low temperatures, when the mineral aggregate remains the same, hardening the asphalt tends to increase stability more markedly than at high temperatures. With regard to the effect of mineral filler, it may be stated that curves at the same temperature for all three mixtures showed increasing stability values as the percentage of filler became greater.

The direct effect of temperature upon the stability of individual mixtures is of very great interest from a practical standpoint and each mixture with its various asphalts was so plotted. Figure 9 shows the relations developed by the extremes and average of the steam-refined asphalt mixtures, and may be considered as typical.

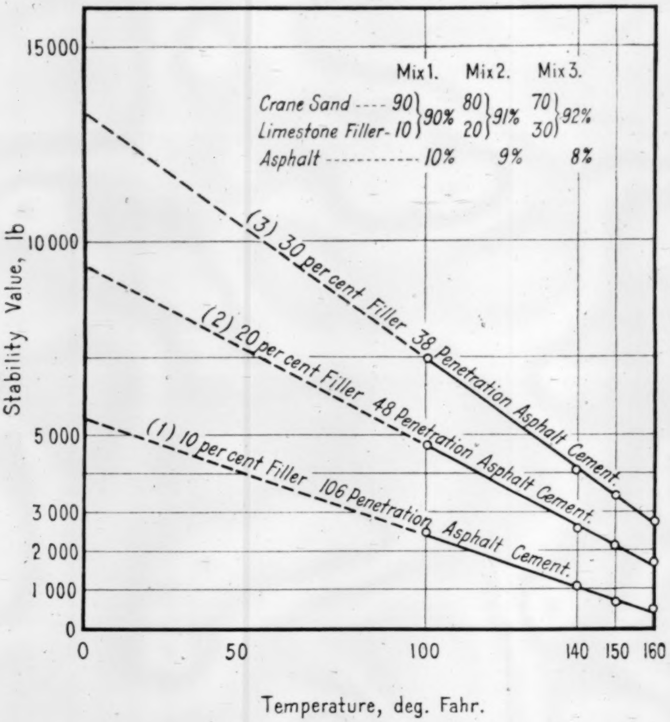

FIG. 9.—Relation of Stability to Temperature.

It is seen in each case that, within the temperature range at which tests were run, practically straight line relations exist and that for a given mix with a given degree of compression, stability is apparently a direct function of temperature of the mix and increases as the temperature decreases. Some variations from this rule were found, possibly due to experimental errors, but so many of the mixtures developed this approximately straight line relation that the existence of a law is fairly well indicated. If further investigation confirms this relation it will make it possible to determine the stability of a given mixture at any temperature by making two determinations at different temperatures and projecting a straight line through the values obtained.

The dotted lines in Fig. 9 are projections of the stability values of the three mixtures to 0° F., at which temperature very wide differences are shown. It is quite well understood in the paving industry that some mixtures, which have proved to be very stable, become so hard and brittle as to crack badly at low temperatures. It is very necessary therefore that in any attempt to increase stability this factor should be kept in mind. If the straight line temperature relation is confirmed a practical means of controlling tendency to crack is at once indicated as a use of the stability test and the authors hope shortly to concentrate on this phase of their investigation. The field is promising as it has already been pointed out that the same degree of stability at a relatively high temperature may be obtained with a soft asphalt and a high percentage of filler as with a harder asphalt and a lower percentage of filler. The temperature slopes of such mixtures are, however, different, so that considerable differences in stability at low temperatures are indicated and it would seem that between two mixtures with the same stability at 140° F. the one showing a slope with the lesser pitch would be the more resistant to cracking.

In recapitulation particular attention is directed to the following deductions which may be drawn from this investigation of factors affecting the stability of asphalt paving mixtures:

1. Closeness of packing or reduction of voids by compression of the mineral aggregate exerts a much greater stabilizing effect than the mere reduction of voids in the entire mixture. Specific gravity or density of the mixture itself is therefore not always a direct indication of either degree of compression or stability.

2. Asphalt films surrounding the particles of compressed mineral aggregates may be thinner than the air or liquid films which exist in the usual methods of determining voids. Therefore, any attempt to proportion asphalt in a mixture with the idea of completely filling the voids as determined in the mineral aggregate may result in the use of too much asphalt with a marked reduction in stability.

3. The greatest stability for a given mineral aggregate has not been obtained with that proportion of asphalt which would produce the lowest percentage of voids in the compressed mixture. The fact that further reduction of voids in the mineral aggregate is likely to occur under traffic after a pavement has been laid, due both to additional compression and internal wear, indicates that from the standpoint of stability, mixtures should be designed so as not to be com-

pletely voidless even after thorough compression during construction. Cases have been noted where pavements have been quite stable for a number of years following construction and have afterwards suddenly become unstable with no sudden change in traffic. It is believed that in such cases while the proportion of asphalt was originally satisfactory for the degree of compaction of the mineral aggregate, a further reduction in its voids under traffic made the quantity of asphalt excessive.

4. In order to secure maximum stability in paving practice the percentage of asphalt must be controlled within much narrower limits for high-filler low-void aggregates than for relatively low-filler high-void aggregates.

5. Of the physical characteristics of asphalt usually determined, penetration at 77° F. appears to exert the most direct and definite effect upon stability of the mixture at all temperatures and a general increase in stability, produced with a given mineral aggregate, accompanies decrease in penetration of the asphalt.

6. Next to closeness of packing of the mineral aggregate, percentage of filler appears to be the greatest stabilizing factor in paving mixtures and provided the proper proportion of asphalt is used, increased stability is invariably produced by increase in filler, at least to the extent that such addition decreases voids in the mineral aggregate. If, however, the proper proportion of asphalt is not used an increase in filler may result in decrease in stability.

7. The stability of a given compressed mixture appears to be directly proportional to its temperature but the proportion varies for different mixtures so that of two mixtures which show the same stability at 140° F. one may be much harder and more likely to crack at low temperatures than the other.

DISCUSSION

Mr. Howe.

MR. HENRY L. HOWE, JR.[1] (*presented in written form*).—I have gone over the paper with much interest, as our laboratory has carried on similar work, on a far less extensive scale, into the characteristics of asphalt paving mixtures. We are mainly concerned, however, in the practical application of the knowledge acquired by such researches in the every-day control of our contractors' asphalt mixing plants, so that my natural inclination in going over such a technical paper is to see how we can apply the knowledge gained to improve such inspection and control.

I believe the procedure should, if possible, be so simplified that the density briquets or cylinders can be made at the mixing plants by the inspectors with the aid of a small hydraulic jack or press equipped with a gage or some pressure-limiting device. Apparent specific gravity or density determinations on such specimens would indicate whether or not the mixtures were of such a character that normal densities could be obtained when compacted on the street. In connection with such specimens made at the plant, the old "pat" or "stain" test would act as a check, in case the density was lower than normal, as to whether the voids in the aggregate had been over-filled with asphalt cement, giving a "fat" stain or mixture and possibly an unstable mixture, or whether insufficient asphalt cement was being used to properly fill the voids giving a "light" stain and a mixture high in voids and of high absorption.

The density of the specimens made at the plant can be compared with actual cylinders cut out of the street by means of the asphalt pavement sampling set developed in our laboratory and described by Messrs. Hubbard and Field in their paper before the American Society of Municipal Improvements last year.

It should be possible to use the authors' stability test apparatus at the plants to check the stability of molded cylinders and those cut from the pavement as laid. Such a test should be very practical provided the straight-line relation between temperature and stability can be proved. However, it is interesting to note that Hubbard and Field found that maximum stability for a given aggregate mixture, as measured by their apparatus, was developed when the mineral

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[1] Director of Municipal Testing Laboratory, City of Rochester Department of Engineering, Rochester, N. Y.

Mr. Howe. aggregate was most closely packed so as to give the minimum aggregate voids in the compressed mixture. Our work indicates that such a point is also the point of maximum practical density or apparent specific gravity for a given mixture and the point beyond which any increase in the percentage of asphalt cement will decrease the stability of the compacted mixture.

If these relations can be proved to be generally true, the fairly simple plant control of the mixture as outlined above, that of making determinations for apparent specific gravity on compacted specimens either made or cut from the street but checked for insufficient or excessive bitumen by means of the "pat" test, should give results that could be depended upon where the density limits for the various mixtures are set by the laboratory and based upon proper analysis and tests.

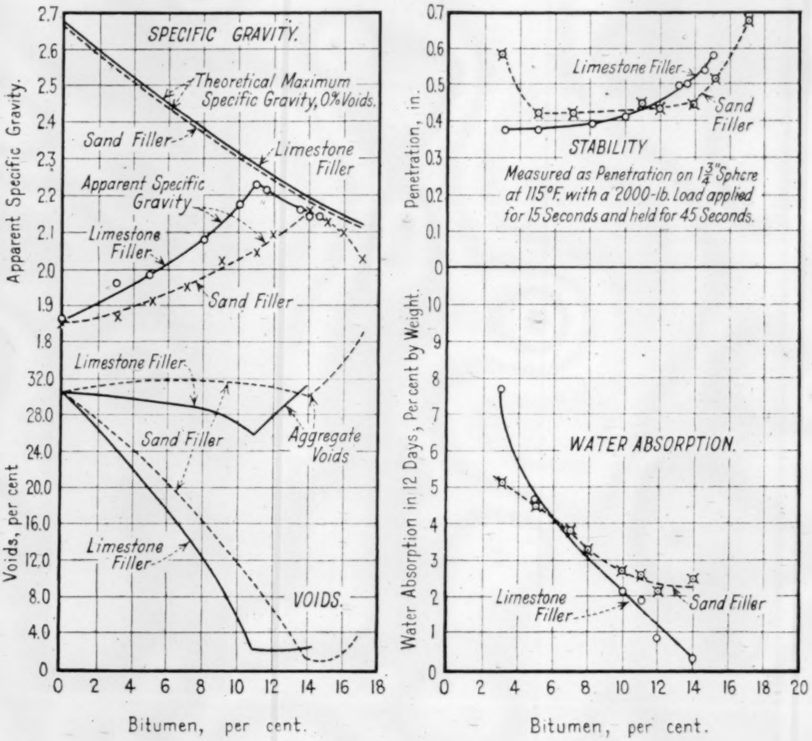

FIG. 1.

I agree with Mr. Hubbard that any general attempt to set the maximum allowable amount of asphalt cement to be added to a pavement mixture by determinations of the voids in the aggregate alone

Mr. Howe. is apt to lead to the mixture being very "fat" due to the effect of the asphalt cement in reducing the aggregate voids, particularly in mixtures containing very finely ground dust filler.

The reasons for this have been outlined by Mr. Hubbard and can be illustrated by studying the characteristics of a sheet asphalt paving mixture, shown in the accompanying Fig. 1.

It will be noted that the density increases with increase in the percentage of bitumen. Also that the density increases much more rapidly when true ground limestone dust filler is used than when the sand or collector dust filler is used, rising to a maximum in each case as it approaches the theoretical limit for zero voids, and then decreasing after the bitumen has been added to excess—a point for each condition of not quite minimum voids as found by Mr. Hubbard.

Note that this peak of maximum density is also the point of maximum compaction of the aggregate for each condition and the points beyond which any further addition of asphalt cement will cause rapid loss in stability.

Mr. Hubbard finds this point to be also the point of maximum stability when measured by his apparatus which, as he says, measures the initial internal friction of the mixture, whereas the ball penetration test probably measures the resistance to plastic flow and does not show maximum stability at these points.

Mr. Rea. MR. A. S. REA.[1]—I should like to ask Mr. Hubbard if he would consider a mixture of 30 per cent filler and 8 per cent bitumen satisfactory and also whether he has made any impact tests on any bituminous mixtures.

In carrying on some research work in connection with physical tests on rock asphalts, we found that some mixtures which gave excellent results in deformation and compression tests, gave very poor results when subjected to an impact test. I believe that it would be well worth while to make some comparative tests of this nature on bituminous paving mixtures to correlate them with other tests for stability.

Mr. Hubbard MR. PRÉVOST HUBBARD.—I will answer the last question first. We have made no impact tests on any of our mixtures so far. I know of very satisfactory work in which over 20 per cent of filler, equivalent to 20 per cent of 200-mesh material, was used with 8 per cent of asphalt. I should not ordinarily recommend as lean a mixture for use in the North; this work was done in the South. On account of

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[1] Engineer of Tests, Ohio Department of Highways and Public Works, Ohio State University, Columbus, Ohio.

Mr. Hubbard. the tendency of the mixture to crack, a greater percentage of asphalt should be used in cold climates.

I wish to make one point plain. We do not know yet what values to assign to this stability test, that is, what values we should work for. It may be best to work for a range that will be considerably below the maximum stability we can develop by the test. In other words, we may not want to work for the maximum stability obtainable from of a given set of constituents, but we now have a yard stick with which to measure stability, and the most desirable range can be determined only by correlation of test results with the service behavior of pavements which have been subjected to the test. At this time it would be very dangerous in the design of a mixture to work to the highest stability we can possibly get by this test.

Mr. Gage. MR. R. B. GAGE.[1]—Judging from the data shown by Mr. Hubbard, it would appear that as the stability of these mixtures increases their durability will decrease. I may be mistaken in this respect in judging the paper or data presented, for I did not have a copy of the paper to read before the meeting.

The sand used by the authors in making these tests is the same as that from which most of the bituminous pavements have been constructed in the northeastern part of New Jersey. We have used a tremendous quantity of this sand in state highway work and the quantity of filler used is usually from 15 to 20 per cent; consequently, our mixtures are very similar to those used in the experiments described. However, with this same sand and filler mixture, we will use about 11 per cent of asphalt cement which, according to the tests reported in the paper, shows very little stability. This mixture has given us very good results and to date we have had practically no failures with it when the pavements have been properly constructed with no defects in the base. Such a pavement, if constructed with 8 per cent asphalt cement, would certainly have a very limited life. In fact, if constructed with 9 or 9.5 per cent of asphalt, it is very doubtful if it would last five years without showing considerable cracking and perhaps some surface disintegration. Pavements of this kind are only accepted at a reduced price, which is determined by our Penalty Schedule and represents the value of the pavement to us. A pavement with only 8 per cent of asphalt cement would be of little or no value to us and it would be optional whether we would accept it free of cost or compel the contractor to replace it.

It is very doubtful if the word "stability" is the proper one to use in this connection, for what has been determined is "strength" and not stability, since stability as used might be considered to include durability also, which evidently is not the case as used here. **Mr. Gage.**

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[1] Chemical Engineer, New Jersey State Department of Conservation and Development, Trenton, N. J.

It is my belief that what we desire is a mixture that has a certain amount of plasticity or workability when subjected to ordinary traffic conditions in warm weather. This plasticity appears to be necessary for the pavement to re-adjust itself to temperature changes, for it has been noted that pavements constructed with asphalt cements that are very susceptible to temperature changes and have high ductilities are not as prone to crack as those constructed from the inert non-ductile bitumens. If these tests can finally be made to show the plasticity that a paving mixture should have to give best results, it may be found possible that such a plasticity can be secured by the use of many more different gradings of sand and types of mineral fillers than we are now accustomed to using.

Mr. Ulman. Mr. M. H. Ulman.[1]—Mr. Hubbard and Mr. Howe both stated that the stability tests are a partial function of the molding of specimens. We have recently conducted some investigations in collaboration with another laboratory in which the same asphalt cement, sand and filler were used. The asphalt in the mixture was varied from 8 to 15 per cent in increments of 1 per cent, and the filler, which was portland cement, held constant at 12 per cent.

In preparing the cylinders for the various tests, we experimented in the molding by using a mold which had been slightly heated, and one that was maintained at a uniform specified temperature by electric heating. The specific gravity or density of the mixture with 10 per cent of asphalt content made in the electrically heated mold was 2.231, while the specific gravity of the cylinders prepared in the warm mold was 2.180. A difference comparable to the above was obtained in the other mixtures by these two methods in molding up to an asphalt cement content of 12 per cent. The temperature of the electrically heated mold had to be reduced when the mixtures exceeded this amount of asphalt cement.

A difference in density, in consideration of the same load having been used in molding, would naturally cause a difference in the laboratory stability tests. It is therefore essential that a standardized method for molding be used in all tests for stability of bituminous mixtures.

Mr. Smith. Mr. F. P. Smith.[2]—What Mr. Gage has just said I think was really brought out by Mr. Hubbard's presentation of the paper. It

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[1] Assistant Engineer of Materials, Pennsylvania State Highway Department, Harrisburg, Pa.

[2] Member of Firm, Dow and Smith, New York City.

Mr. Smith. appears to me that the chief value of this paper lies in establishing a yard stick by means of which the effect of certain variations in the composition of an asphalt mixture may be measured. The authors, as I understand it, have not taken the position that the maximum stability which they have been able to obtain is necessarily the most desirable or practical thing to effect in a mixture. There is no question but that a low-bitumen high-filler mixture carrying 30 per cent of filler is very stable, but its stability is greatly affected by minute variations in the bitumen content of the mixture and it is extremely doubtful whether it would be practical to turn out such a mixture with the required degree of uniformity from an ordinary paving plant. In the case of a mixture which contains only 10 per cent of filler, we find that much greater variations in bitumen are permissible without seriously affecting the stability of the mixture and, all things considered, it is probable that the most practical mixture lies somewhere between these two extremes.

Another point which has to be taken into consideration in interpreting the results is the difficulty of compressing some of these high-filler low-bitumen mixtures. It is my personal experience that mixtures of that type are so difficult to compress upon the street satisfactorily that more is lost than gained, in trying to lay them, although theoretically and under perfect conditions enormously high stability results can be obtained with mixtures of that type.

Personally, I want to add my note of commendation to Mr. Hubbard's comments on the undesirability of limiting or determining the amount of bitumen desirable in a mixture on the determination of the void contents of the mineral aggregate alone. My own experience is strongly corroborative of Mr. Hubbard's remarks on that subject.

There is one other point to which I should like to call attention for a moment—Mr. Gage mentioned it—and that is, the low-bitumen mixtures of high stability are extremely susceptible to cracking in cold weather and even in moderate temperatures, where the traffic is light, and this should not be lost sight of when considering mixtures of this sort from the practical service standpoint. Our experience in the past has been that low-bitumen mixtures are very susceptible to cracking, more especially where asphalt cements of medium or low penetration have been used, and also more especially in those cases where the traffic is light. There are many examples of pavements throughout this country, containing 8.5 to 9 per cent of bitumen, which have cracked very badly.

Mr. Smith. The authors fully comprehend, I take it, from the statements made in the paper, that exactly what the test really measures is a subject that is, perhaps, open to very wide discussion. The phenomenon of shoving or pushing of a pavement under traffic frequently occurs a number of years after the pavement has been laid. It is quite possible that the stability of the mixture at the time it shoves has become lessened, through some cause or other, to a point below what it originally possessed. This lessening of stability may have been due to the continuous stress on the coating films of bitumen caused by the motion of the sand grains under the kneading action of traffic, or it may have been due to the lessening of the cementing value of the bitumen due to exposure and aging, or it may have been due to other causes. The authors' test has measured, so far as the results presented are concerned, the stability of a mixture immediately after the manufacture and compression of that mixture. It would be a very interesting development, indeed, to investigate how far those results are comparable with results on the same mixture after it has been subjected to the kneading effect of traffic and after a considerable time has elapsed.

Mr. Hubbard MR. HUBBARD.—In closing, I might say that we have quite carefully studied the particular point Mr. Ulman mentioned. Mr. Field made a large number of tests upon the same mixture compressed under different conditions, varying the original temperature of mix, temperature of mold, and method of compression, and as a result of this work has developed a detailed method which is described in our paper.

A later development is the devising of a special tamper which first tamps the outside area of the testing face and then the inside area before direct compression is put on the specimen. In this way we obtain to some extent a duplication of the kneading action of a roller. At any rate, we get a greater density, and we find that as we work in these little refinements we are getting to the point where the maximum practical density is obtained quite readily.

EFFECT OF SIZE OF PAVING BRICK ON RATTLER LOSS

By F. H. Jackson[1]

Synopsis

This paper gives the results of a series of tests made at the U. S. Bureau of Public Roads to determine the effect of size or volume of paving brick on the rattler loss. A series of five different sizes of brick from a single plant were tested in the rattler and a correction curve derived from the results obtained, which it is believed may be used in establishing a series of correction factors for use in specifications.

It has long been recognized that for brick of equal quality but differing in size the comparative rattler losses are not directly proportional to the differences in weight. Therefore, any system of rating based on the percentage of loss by weight, irrespective of the difference in size, is incorrect unless a correction is introduced covering this feature or unless independent standards are set up for each size separately.

In the very elaborate series of tests presented by Blair and Orton before the American Society for Testing Materials in 1911,[2] upon which our present standard method of test is largely based, no mention is made of the effect of size on rattler loss. This was probably due to the fact that at that time most of the paving brick manufactured was of the so-called "standard block" size, that is, about 3 to 3½ in. in width, 4 in. in depth, and 8 to 9 in. in length. Talbot, however, in his paper on "Qualities of High-Grade Paving Brick,"[3] states that, although he has not studied the effect of the size of brick on the rattler loss, "it is established that the brick size will sustain a greater loss than the block size of the same grade and quality. . . . The amount of this difference depends upon various conditions, but with good material the brick sizes may be expected to lose, say, 3 per cent more than the block sizes."

It has only been within comparatively recent years that the thinner brick, such as those made to lay to a depth of 3 in. and 2½ in., have come into general use. Efforts have also been made from time to time to introduce certain odd sizes, such as the 3 by $3\frac{1}{2}$ by $8\frac{1}{2}$ in., and others in which the length varied slightly. The number of sizes of brick in use, however, has been greatly curtailed within the last three years through the efforts of the permanent committee on the elimination of unnecessary sizes and types of paving brick, sponsored by the U. S. Department of Commerce. At the present time this committee recognizes two sizes of brick, as follows: $3\frac{1}{2}$ by 4 by $8\frac{1}{2}$ in., and 3 by 4 by $8\frac{1}{2}$ in. In addition to these two sizes, the $2\frac{1}{2}$ by 4 by $8\frac{1}{2}$-in. size is coming into rather general use, so that it will in all probability in the near future be included in the series of recognized sizes.

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[1] Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[2] M. W. Blair and Edward Orton, Jr., "A Study of the Rattler Test for Paving Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 776 (1911).

[3] Illinois State Geological Survey Bulletin 9, "Paving Brick and Paving Brick Clays of Illinois."

Recognizing the injustice of specifying the same percentage of wear for both 3-in. and $3\frac{1}{2}$-in. brick, many paving engineers, when they began using the thinner brick for construction, adopted the practice of inserting certain arbitrary correction factors in their specifications so as to bring these sizes into line with the requirements for the so-called "standard block" size. So far as the author is aware, however, none of these correction factors was based upon extensive test data. As a rule, they were the result of theoretical consideration.

In view of the fact that Committee C-3 on Brick of the American Society for Testing Materials has undertaken to rearrange the Standard Specifications for Paving Brick (C 7 – 15), in order to bring them into conformity with the Society's present standards as to form, it seemed an excellent opportunity to investigate this relationship experimentally with a view to furnishing a table of correction factors which might be inserted in the standard.

TESTS MADE ON FIVE SIZES OF BRICK

Fortunately, a rather unusual opportunity existed for obtaining such data. The U. S. Bureau of Public Roads has had under way for the past several months an investigation to determine the relation between the depth of the paving brick wearing course and the resistance of the pavement to the action of traffic. This investigation is being conducted by the Bureau in cooperation with the National Paving Brick Manufacturers Association, which furnished a large quantity of brick for this purpose. These brick were all from the same plant and were manufactured as a special lot so as to be as nearly uniform in quality as possible. They were of the plain wire-cut type, and the average rattler loss on the $3\frac{1}{2}$ by 4 by $8\frac{1}{2}$-in. size was about 17.0 per cent. Five sizes of brick were furnished, all of the same width and length but varying in depth from 2 to 4 in. in $\frac{1}{2}$-in. steps.

It was decided, therefore, in addition to the major investigation, to make an incidental study of the effect of size on rattler loss, using the 5 sizes of brick on hand. For this purpose 10 standard rattler tests were made on each size of brick, making 50 tests in all. Every precaution was taken to keep the standard rattler calibrated, and every detail as called for in standard procedure was followed. The results for the first series of rattler tests are given in Table I. Upon studying the results it became immediately apparent that there was no constant relation between the average percentage of loss and the size of the brick. This at once suggested the possibility that the various sizes of brick might not all be of the same quality. In order to throw light upon this important point, a number of brick of each size were subjected to a special hardness test, using the Dorry hardness machine for testing rock. One-inch cores were drilled with a diamond drill from the center of each brick and subjected to the abrasive action of quartz sand, fed upon a revolving steel disk upon which the brick core was held under a standard pressure. The loss in weight of the specimen at 2500 revolutions of the disk was considered to be a true measure of its hardness.

TABLE I.—RATTLER TEST RESULTS ON BRICK USED TO DETERMINE EFFECT OF SIZE.

Sample	Loss in Weight, per cent, for Brick of Various Thicknesses				
	2 in.	2½ in.	3 in.	3½ in.	4 in.
No. 1	22.6	17.9	18.6	17.4	17.3
No. 2	22.0	21.4	18.3	16.8	16.5
No. 3	23.1	18.3	19.7	16.8	16.4
No. 4	21.9	18.3	19.7	16.5	16.3
No. 5	22.8	19.1	18.7	16.2	17.0
No. 6	22.8	19.3	19.7	17.0	17.1
No. 7	22.7	19.0	18.6	17.2	16.3
No. 8	22.9	17.6	19.3	17.0	17.2
No. 9	24.7	19.2	19.1	18.0	17.4
No. 10	23.3	17.9	18.2	17.4	16.6
Average	22.9	18.8	19.0	17.0	16.8
Maximum	24.7	21.4	19.7	18.0	17.4
Minimum	21.9	17.6	18.2	16.2	16.3

Realizing that hardness is not the only quality of a paving brick which affects the rattler loss, tests for crushing strength and transverse strength were also made. The results of these tests, together with the results of the hardness tests, are plotted in Fig. 1. Crushing strengths were determined on half brick, tested on edge, five tests of each size being made. The brick were bedded in plaster of Paris before testing. Tests for transverse strength were made in two ways, first, by using a special form of equalizer apparatus developed at the Bureau of Standards, and second, by the use of A.S.T.M. standard apparatus[1] somewhat modified by the Bureau of Public Roads. Details of the results of the transverse tests, discussed from the standpoint of comparative methods of testing, will be published elsewhere. For the purpose of this discussion, the results of the tests by both methods were averaged. Each average is the result of 40 tests.

A very brief study of these tests is in order with a view to determining what differences in quality exist between the various sizes. It will be seen at once that the tests are not altogether consistent. For instance, the 4-in. size has a considerably higher crushing strength than any of the other sizes. This difference, however, is not reflected in any of the other tests, which is, of course, not surprising when we consider that each of these tests measures a specific property of the brick. When taken as a whole, however, the results give a general idea of the relative quality of the five sizes under consideration which, in this case, is what we are after. Assuming for the moment that the resistance of brick in the rattler is influenced by both hardness and toughness and that these qualities are measured individually probably better by the hardness test and the transverse test than by any of the others, we find that from the standpoint of hardness the 2-in. brick

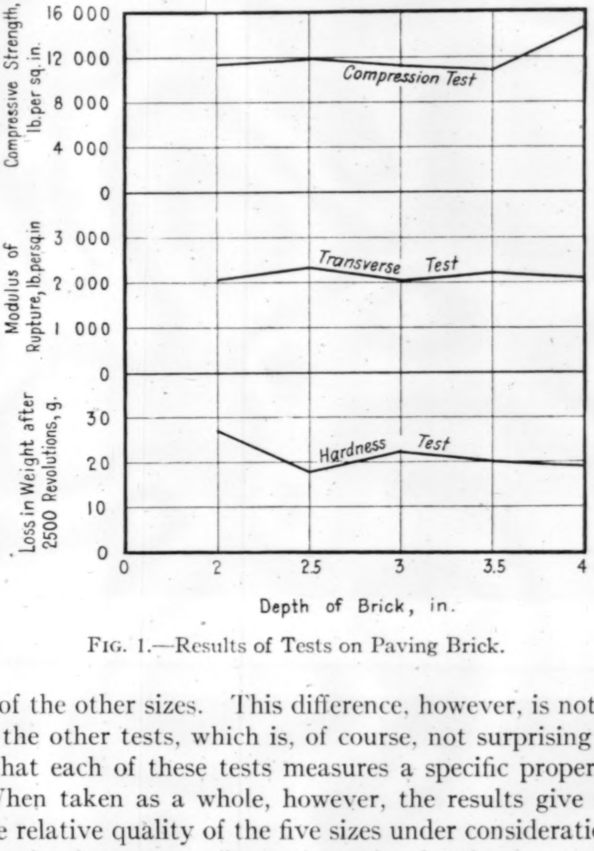

FIG. 1.—Results of Tests on Paving Brick.

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[1] 1924 Book of A.S.T.M. Standards, p. 665.

are considerably softer than any of the other sizes; the 3-in. are next; and the 2½-in. are the hardest. In transverse strength the 2½-in. size ranks highest; the 3½-in. next; with the 2-in., 3-in. and 4-in. practically identical. Taking both tests into consideration, we may tentatively rate the brick relatively as to quality about as follows: 2½-in., 3½-in., 4-in., 3-in., 2-in. It should be borne in mind, of course, that these differences are not large numerically, and are of significance only because of the special use to which the rattler tests will now be put.

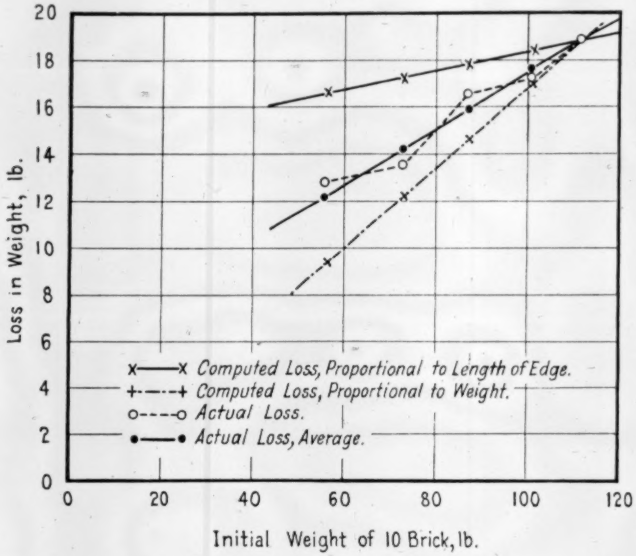

FIG. 2.—Relation Between Weight of Brick and Rattler Loss.

EFFECT OF SIZE ON RATTLER LOSS

In Fig. 2 are plotted the average losses in pounds for each size of brick against the initial weight of the brick charge. There are also plotted two series of points, one above and the other below the actual losses, which show what the losses would have been if they had been (1) directly proportional to differences in the number of linear inches of edge exposed to wear, and (2) directly proportional to differences in weight (or volume). It will be seen that the actual curve is somewhat below a line bisecting the angle formed by the two theoretical curves, which indicates that the correction to be applied to the small sizes is somewhat smaller than has been commonly used on the assumption that the loss takes place principally on the edges and corners of the brick.

Returning now to a discussion of the actual losses, we find that the plotted points do not lie on a straight line as they would were the brick all of the same quality and the differences in loss due entirely to the effect of size. Plotting the average line, we find that the losses for the $2\frac{1}{2}$-in. and $3\frac{1}{2}$-in. sizes lie below the line, while those for the 2-in. and 3-in. sizes lie above the line. This grouping is exactly what would be expected as a result of our study of the relative quality of the five sizes based on the hardness and transverse tests, and indicates that these tests are a reliable measure of those properties of the paving brick which are affected by the rattler test.

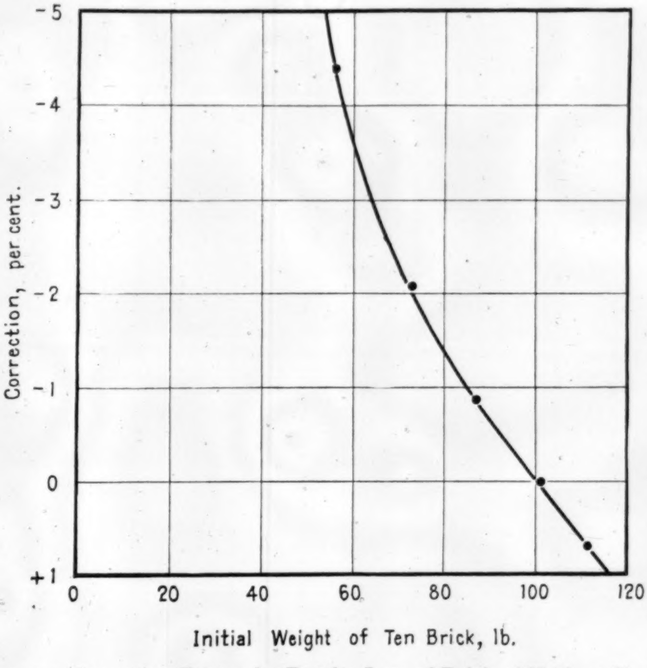

FIG. 3.—Correction Factor for Rattler Loss of Brick of Various Size.

We would seem justified, therefore, in drawing the average line as indicated on the chart, and recomputing the various percentages of loss from the corrected losses obtained therefrom. This gives us a series of values the same as would have been obtained experimentally had all the brick been of the same quality. Using these values, a correction curve has been plotted as shown in Fig. 3. By means of the chart it is possible to determine what percentage shall be added to or subtracted from the observed rattler loss to give the equivalent value in terms of a "standard" 10-lb. brick. The corrections, it will be observed, are based entirely on weight and not on nominal size. While it might be more convenient to use the latter method, it would seem unwise to do so because of the wide variations in actual size of brick of the same nominal size. For instance, among the 8 brands tested by the bureau in connection with this work, it was found that for the 2½-in. size the actual weights of 10 brick varied from 64 to 75 lb., while for the 3-in. size the corresponding variation was from 82 to 89 lb. These differences, of course, are accounted for in part by differences in specific gravity of the material. In general, however, the differences in size appear to be more pronounced as will be observed by noting Fig. 4, in which are plotted the average weights and corresponding volumes for each of the brands and sizes of brick tested.

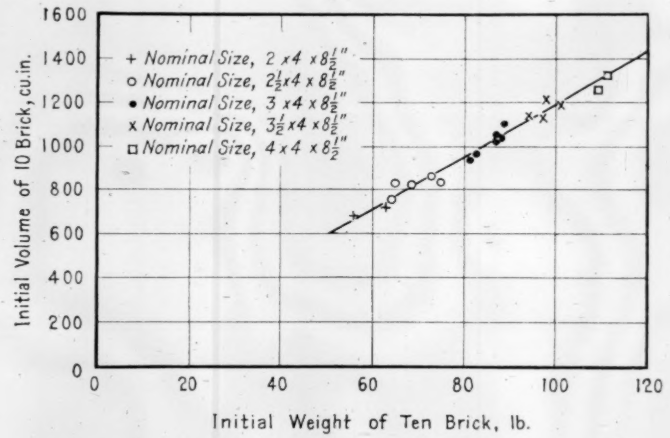

FIG. 4.—Relation Between Initial Volume and Weight of Brick.

It will be observed that the points lie fairly well on a straight line, those above the line indicating the lighter brick, while those below the line indicate the heavier brick. Of course, a correction based on the volume of brick would be the most rational method because it would eliminate variations in the volume-weight relations due to differences in specific gravity. However, the weight determination is much simpler and more readily made, and it would not appear that the small changes in specific gravity which normally occur in well-burned paving brick would seriously affect the accuracy of the corrections.

SUPPLEMENTARY CHECK TESTS

In order to determine to what extent the proposed correction curve could be applied in actual work, a series of check tests was run on a number of different brands of paving brick submitted by the manufacturers at the request of the National Paving Brick Manufacturers Association. Each brand was represented by at least two sizes, and in some cases by three sizes. Control tests for hardness and for modulus of rupture were likewise run, in order to check the quality of the brick by tests independent of the size factor. Unfortunately, the number of brick available for these supplementary tests was so limited that it was found impossible to secure a sufficient number of tests for hardness and modulus of rupture to obtain representative averages.

TABLE II.—CHECK TESTS OF PAVING BRICK.

Lot	Nominal Size, in.	Volume of 10 Brick, cu. in.	Weight of 10 Brick, lb.	Rattler Loss, per cent	Corrected Loss, per cent	Total Variations Within Lots, per cent	
						Before Correction	After Correction
No. 1.........	3	1 009	87	24.2	23.3		
	2	720	63	28.1	24.8	3.9	1.5
No. 2.........	3½	1 219	98	19.9	19.8		
	3	1 113	89	21.3	20.6	1.4	0.8
No. 3.........	3½	1 128	97	16.1	15.9		
	3	1 028	87	16.7	15.8		
	2½	838	75	17.8	15.9	1.7	0.1
No. 4.........	4	1 266	109	17.0	17.5		
	3	974	83	17.9	16.7		
	2½	762	64	18.3	15.2	1.3	2.3
No. 5.........	3½	1 141	94	17.1	16.8		
	2½	831	65	21.0	18.0	3.9	1.2
No. 6.........	3½	1 156	94	19.7	19.3		
	2½	811	64	23.2	20.1	3.5	0.8

The results of the rattler tests on 6 lots of brick, representing 4 brands of plain, wire-cut-lug, shale brick and two brands of plain, wire-cut, fire-clay brick are shown in Table II, together with their initial volumes and weights and the corrected percentage of loss derived from the use of the correction curve shown in Fig. 3. Each value for percentage of wear represents the average of three tests. It will be observed that in all cases but one the differences in percentage between the corrected losses for the different sizes in any given lot are considerably less than the corresponding differences before correction. In only one case, however,—that of lot 3—have these differences entirely disappeared, indicating that a certain amount of the original variation was due to differences in the quality of the brick. This is not surprising when it is remembered that no special effort in any case was made to obtain brick of exactly the same quality. In one case—that of lot 4—the results of the individual rattler tests were very erratic, indicating an extremely non-uniform product, which may account for the wide variations of the results obtained on this lot.

It is felt that the correction curve obtained as a result of this series of test is sufficiently accurate for all practical purposes; and the author has recommended that a table of correction factors based thereon be considered for use in connection with any revision of the present standard rattler test for paving brick. The following addition to Section 13 of the Standard Specifications for Paving Brick (C 7 – 15)[1] has been suggested.

"The result obtained in the original calculation shall be corrected by adding to or subtracting from it a factor depending upon the initial weight of the brick charge in pounds. This factor shall be obtained from the following table:

Original Weight of 10 Brick, lb.	Correction to be Applied to Percentage of Wear Obtained by Test, per cent
105 to 115	+0.5
95 to 104	0.0
90 to 94	−0.5
85 to 89	−1.0
80 to 84	−1.5
75 to 79	−2.0
70 to 74	−2.5
65 to 69	−3.0
60 to 64	−3.5
55 to 59	−4.0

"The final corrected value as well as the original value and the correction factor shall be reported."

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[1] 1924 Book of A.S.T.M. Standards.

DETERMINATION OF PRESSURE DISTRIBUTION ON CIRCULAR PIPE WHEN TESTED IN THE A.S.T.M. STANDARD SAND BEARINGS[1]

By Dalton G. Miller[2] and Paul C. McGrew[3]

Synopsis

Results of a series of tests are presented in this paper which show the distribution of pressure on both top and bottom quadrants of circular pipe when under load in the A.S.T.M. standard sand bearings as specified for testing sewer pipe and drain tile. The distribution of pressure has been determined by measuring the starting pull required to move steel strips in canvas laid on the outer circumference of circular pipe and between the pipe and the sand of the bearings. Conclusions are based on the law of physics that for specific materials the starting force required to overcome starting friction is proportional to the load.

Except under most favorable conditions the concentration of loading on the bottom quadrant of pipe tested in the sand bearings is somewhat greater than heretofore assumed while under unfavorable conditions it may be very much greater. Bedding with loose sand containing 2.5 and 5 per cent of moisture produced the most favorable conditions, in the matter of pressure distribution, while bedding with thoroughly compacted dry sand produced the most severe. Considering all degrees of compactness of sand used in the tests, that with 5 per cent of moisture gave the most consistent results.

It is recommended, when applying the crushing test in the A.S.T.M. standard sand bearings, that the sand used contain about 5 per cent of moisture and that the sand of the lower bearing be loosened by spading immediately before placing each tile for the test.

Crushing tests made in the cooperative Drain Tile Laboratory at University Farm, St. Paul, Minn., of 1750 drain tile of sizes 4 to 30 in. brought out discrepancies that apparently indicated that the trouble was due to irregularities in the test results obtained when using the A.S.T.M. standard sand bearings as specified for drain tile and sewer pipe.[4] By a process of elimination the conclusion was finally reached that a greater difference in test results was possible with the standard sand bearings than was generally appreciated, and furthermore that the cause was due apparently to the condition of the sand, used in making the tests, in the matter of compactness and moisture content. Working along these lines, the method described was developed for measuring the distribution of pressure on the quadrants of the tile in contact with the sand of the bearings. The data of these tests are presented in this paper in detail that they may be available for consideration by engineers interested in the testing of circular drain tile, sewer pipe and culverts.

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[1] University of Minnesota *Paper No. 623* Journal Series. This paper is the result of experiments at University Farm, St. Paul, Minn., in the Drain Tile Laboratory conducted by the Department of Agriculture of the University of Minnesota, the Department of Drainage and Waters of the State of Minnesota and the U. S. Department of Agriculture.

[2] Drainage Engineer, Bureau of Public Roads, U. S. Department of Agriculture.

[3] Junior Drainage Engineer, Bureau of Public Roads, U. S. Department of Agriculture.

[4] See 1924 Book of A.S.T.M. Standards, pp. 677, 688, 699.

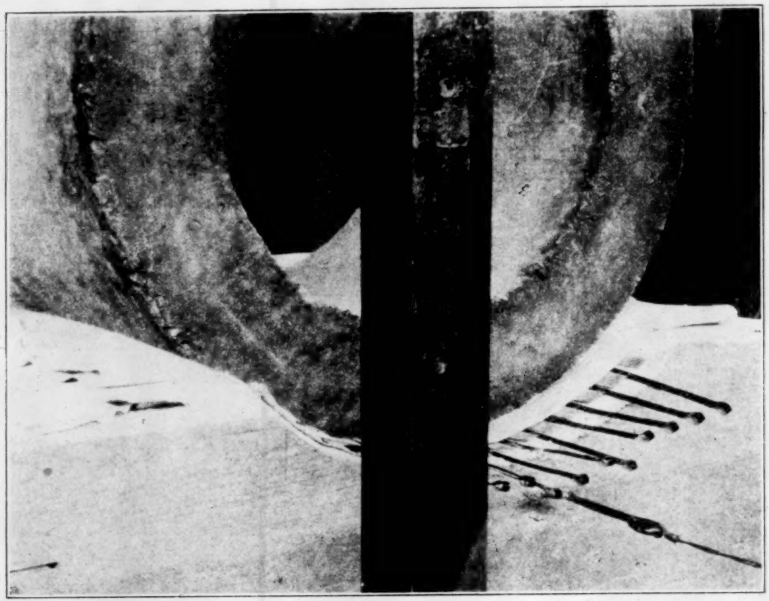

FIG. 1.—Showing Steel Strips in Place as Used in Determining the Pressure Distribution in the Lower Bearing of the A.S.T.M. Standard Sand Bearing.

METHOD USED

The method used in determining the distribution of pressure is based on a well-known law of physics that for specific materials the starting force required to overcome starting friction is proportional to the load. This law of physics was applied, in this work, by measuring in pounds the starting pull required to move steel strips laid between pieces of 10-oz. canvas placed between the sand of the bearings and the outer circumference of a circular pipe of 26-in. outside diameter as illustrated in Figs. 1 and 2. The purpose of the canvas was to provide smooth surfaces over which the friction would remain constant throughout the test. This is a condition that was not possible to realize by laying the strips directly in the sand, as enough sand frequently was displaced when a strip was started as not only to cause erratic check readings of the same strip but also to render readings of adjacent strips very unreliable. A thin rubber sheet was laid between the canvas and the sand to prevent moisture from the sand working into the canvas and thereby changing the coefficient of friction.

In the studies made with the 22-in. pipe, wall thickness 2 in., fifteen steel strips $\frac{5}{16}$ in. in width and $\frac{1}{64}$ in. thick were used. It was found that in order in all cases to get consistent results it was necessary that the area between the strips be covered as nearly as possible by auxiliary strips of the same thickness as the ones pulled. This was required as otherwise when the sand became compacted beyond a certain degree the strips took a proportion of the load in excess of that calculated for their combined areas. The auxiliary strips could have been omitted and the same thing accomplished either by pulling more strips of the same width or by pulling wider ones but the method adopted was the most convenient.

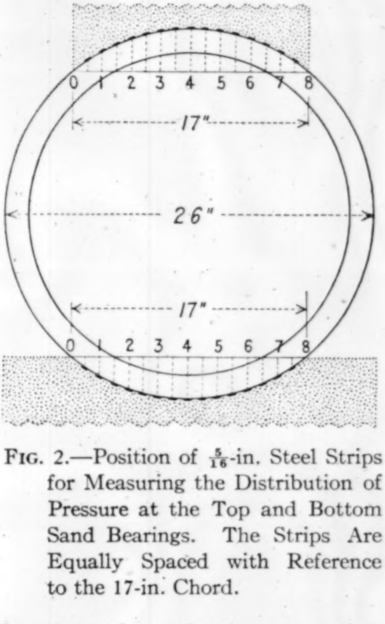

FIG. 2.—Position of $\frac{5}{16}$-in. Steel Strips for Measuring the Distribution of Pressure at the Top and Bottom Sand Bearings. The Strips Are Equally Spaced with Reference to the 17-in. Chord.

The pull in pounds on each strip was measured by means of a spring balance working through a lever arm with a 1:5 ratio. By using the lever arm rather than pulling direct it was possible to control the tendency of a strip to jump an inch or two when started while a single strip could in this way be started, and the scale consistently read, as many as 30 times without more than a slight displacement of the strip.

The 15 strips were equally spaced as indicated in Fig. 2, with reference to the 17-in. cord, $1\frac{1}{16}$ in. center to center. The two end strips, however, were $1\frac{1}{16}$ in. from the extremities of this cord so that the actual area, on the cord, represented by each end strip is 50 per cent in excess of that of the other strips. The load on this excess area, coming as it does at the edge of the sand, was too small to measure and in the calculations has been neglected.

ACCURACY OF RESULTS

The accuracy of any set of readings recorded in Tables I and II may be checked by comparing total pulls for the same loading. Checking in this way, regardless of differences in top and bottom bearings and of bedding conditions due to variations in moisture and compactness of the sand, the greatest departure from the mean total pull has been 5.1, 5.5 and 7.5 per cent, respectively, for loadings of 1680, 2500 and 3500 lb. per linear foot while in general the results have checked within 3 per cent of the average. When the averages of the 14 readings recorded for each of the three loadings in Tables I and II are compared on the basis of the load ratios the results check within 0.5 per cent. There are also, of course, other ways by which the results may be checked, if it is so desired, but in view of the several variables introduced in the tests it is believed that it would be unreasonable to expect greater accuracy than the checks applied indicate.

DISCUSSION OF DATA AND TESTS

In Tables I and II, in the third line of each loading under the various bedding conditions, are recorded, as calculated from the pull on the strips, the actual load reduced to loads on sections, corresponding to respective strips, per linear inch of tile. The pressure has been assumed as vertical on the tile without at this time attempting to resolve any components which may exist. These are the data upon which the graphs of Figs. 3 and 4 are based, the loads being reduced, in the figures, to pressure in pounds per square inch. Note that this pressure is with reference to the horizontal projection of the quadrant of the pipe in contact with the sand as represented by the 17-in. chord of Fig. 2.

A study of the graphs of Fig. 3 brings out several features not heretofore known which affect the distribution of pressure in the lower sand bearing. Explanatory of the three types of beddings considered

TABLE I.—DISTRIBUTION OF PRESSURE IN THE LOWER SAND BEARING OF A 22-IN. TILE UNDER LOADS OF 1680, 2500 AND 3500 LB. PER LINEAR FOOT.

Under each loading the first line is the average pull of two independent sets of readings, the second line is the average of corresponding readings on the two sides of the center strip, while the data in the third line are those of line 2 reduced to loads on sections, corresponding to respective strips, per linear inch of tile. Each section, therefore, measures 1 by $1\frac{1}{16}$ in.

ROOM DRY SAND (0.35 PER CENT MOISTURE)

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Total
							BEDDING NO. 1(A) LOOSE SAND									
1680	1.25	6.75	8.75	10.62	15.62	27.50	28.75	40.63	26.25	28.37	18.75	11.88	10.00	4.75	0.13	240.00
	0.69	5.75	9.37	11.25	17.18	27.94	27.50	40.63	27.50	27.94	17.18	11.25	9.37	5.75	0.69	239.99
	0.40	3.36	5.45	6.57	10.02	16.30	16.05	23.70	16.05	16.30	10.02	6.57	5.45	3.36	0.40	140.00
2500	2.75	8.74	13.75	18.13	26.25	40.00	40.00	55.00	38.13	40.00	28.75	20.00	16.25	6.88	0.25	354.89
	1.50	7.82	15.00	19.07	27.50	40.00	39.06	55.00	39.06	40.00	27.50	19.07	15.00	7.82	1.50	354.90
	0.88	4.59	8.81	11.19	16.15	23.48	22.93	32.29	22.93	23.48	16.15	11.19	8.81	4.59	0.88	208.34
3500	4.75	15.63	20.00	26.25	37.50	53.13	53.13	73.75	49.37	53.13	39.37	28.75	22.13	11.25	0.50	488.64
	2.62	13.44	21.06	27.50	38.44	53.13	51.25	73.75	51.25	53.13	38.44	27.50	21.06	13.44	2.62	488.63
	1.56	8.02	12.57	16.41	22.94	31.71	30.59	44.02	30.59	31.71	22.94	16.41	12.57	8.02	1.56	291.62
							BEDDING NO. 2(A) SLIGHTLY COMPACTED SAND									
1680	0.62	5.25	10.00	8.75	12.50	20.63	45.62	49.37	32.50	23.13	12.13	14.37	10.00	6.88	3.75	255.50
	2.19	6.07	10.00	11.56	12.32	21.88	39.06	49.37	39.06	21.88	12.32	11.56	10.00	6.07	2.19	255.53
	1.20	3.33	5.48	6.33	6.75	11.99	21.40	27.06	21.40	11.99	6.75	6.33	5.48	3.33	1.20	140.02
2500	2.25	10.00	18.75	17.50	22.13	30.00	61.88	65.00	41.88	32.50	20.00	21.25	15.63	11.62	5.63	376.02
	3.94	10.81	17.19	19.37	21.07	31.25	51.88	65.00	51.88	31.25	21.07	19.37	17.19	10.81	3.94	376.02
	2.18	5.99	9.52	10.73	11.67	17.31	28.74	36.01	28.74	17.31	11.67	10.73	9.52	5.99	2.18	208.29
3500	5.25	18.75	30.62	25.13	35.63	43.75	81.25	85.00	56.25	46.25	28.75	30.63	22.13	16.88	10.00	536.27
	7.63	17.82	26.37	27.88	32.19	45.00	68.75	85.00	68.75	45.00	32.19	27.88	26.37	17.82	7.63	536.28
	4.15	9.69	14.34	15.16	17.51	24.47	37.39	46.23	37.39	24.47	17.51	15.16	14.34	9.69	4.15	291.65
							BEDDING NO. 3(A) THOROUGHLY COMPACTED SAND									
1680	0.75	4.75	9.37	10.00	10.62	25.00	48.13	63.75	47.50	22.50	8.75	6.00	3.87	1.88	0.63	263.50
	0.69	3.32	6.62	8.00	9.68	23.75	47.82	63.75	47.82	23.75	9.68	8.00	6.62	3.32	0.69	263.51
	0.37	1.76	3.52	4.25	5.14	12.62	25.41	33.87	25.41	12.62	5.14	4.25	3.52	1.76	0.37	140.01
2500	2.12	7.62	15.62	17.50	17.50	38.13	68.12	81.88	66.25	35.00	16.25	7.50	6.00	3.50	1.63	384.62
	1.88	5.56	10.81	12.50	16.88	36.57	67.18	81.88	67.18	36.57	16.88	12.50	10.81	5.56	1.88	384.64
	1.02	3.01	5.86	6.77	9.14	19.81	36.39	44.35	36.39	19.81	9.14	6.77	5.86	3.01	1.02	208.35
3500	4.00	11.25	24.13	27.50	28.75	52.50	85.62	101.25	86.25	48.75	26.25	10.62	8.75	6.75	3.00	525.37
	3.50	9.00	16.44	19.06	27.50	50.63	85.94	101.25	85.94	50.63	27.50	19.06	16.44	9.00	3.50	525.39
	1.94	5.00	9.13	10.58	15.27	28.11	47.71	56.21	47.71	28.11	15.27	10.58	9.13	5.00	1.94	291.69

SAND CONTAINING 2.5 PER CENT MOISTURE

BEDDING NO. 1(B) LOOSE SAND

Load on Tile, lb. per linear ft.	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Total
1680	8.00	10.62	14.37	17.50	21.25	18.75	24.37	24.37	24.12	23.13	20.63	15.62	8.75	7.62	5.50	244.60
	6.75	9.12	11.56	16.56	20.94	20.94	24.25	24.37	24.25	20.94	20.94	16.56	11.56	9.12	6.75	244.61
	3.86	5.22	6.62	9.48	11.98	11.98	13.88	13.95	13.88	11.98	11.98	9.48	6.62	5.22	3.86	139.99
2500	11.25	18.75	22.50	25.00	31.88	28.13	36.88	36.88	35.62	36.25	31.88	24.37	13.37	11.25	8.25	372.26
	9.75	15.00	17.94	24.68	31.88	32.19	36.25	36.88	36.25	32.19	31.88	24.68	17.94	15.00	9.75	372.26
	5.46	8.39	10.04	13.81	17.84	18.01	20.29	20.64	20.29	18.01	17.84	13.81	10.04	8.39	5.46	208.32
3500	16.25	25.63	32.50	35.00	45.62	39.37	52.50	51.88	51.25	50.63	46.25	35.63	21.25	18.13	11.88	533.77
	14.06	21.88	26.88	35.32	45.94	45.00	51.88	51.88	51.88	45.00	45.94	35.32	26.88	21.88	14.06	533.80
	7.68	11.95	14.69	19.30	25.10	24.59	28.35	28.35	28.35	24.59	25.10	19.30	14.69	11.95	7.68	291.67

TABLE I.—DISTRIBUTION OF PRESSURE IN THE LOWER SAND BEARING OF A 22-IN. TILE UNDER LOADS OF 1680, 2500 AND 3500 LB. PER LINEAR FOOT (*Continued*).

SAND CONTAINING 2.5 PER CENT MOISTURE (*Continued*)

BEDDING NO. 2(B) SLIGHTLY COMPACTED SAND

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
1680	0.62	1.25	3.50	13.13	22.50	24.37	41.25	46.25	33.75	21.88	19.37	16.25	4.50	1.50	0.88	251.00
	0.75	1.38	4.00	14.69	20.94	23.12	37.50	46.25	37.50	23.12	20.94	14.69	4.00	1.38	0.75	251.01
	0.42	0.77	2.23	8.19	11.68	12.90	20.92	25.80	20.92	12.90	11.68	8.19	2.23	0.77	0.42	140.02
2500	0.88	3.63	6.75	22.50	36.25	38.75	55.62	60.63	46.88	34.37	28.75	24.37	7.38	3.00	2.25	372.01
	1.57	3.31	7.06	23.44	32.50	36.56	51.25	60.63	51.25	36.56	32.50	23.44	7.06	3.31	1.57	372.01
	0.88	1.85	3.95	13.13	18.20	20.47	28.70	33.95	28.70	20.47	18.20	13.13	3.95	1.85	0.88	208.31
3500	1.75	7.75	11.88	37.50	51.88	53.13	66.88	70.00	60.63	48.75	43.75	38.75	13.75	6.37	4.50	517.27
	3.13	7.06	12.81	38.13	47.82	50.94	63.75	70.00	63.75	50.94	47.82	38.13	12.81	7.06	3.13	517.28
	1.76	3.98	7.22	21.50	26.96	28.72	35.94	39.47	35.94	28.72	26.96	21.50	7.22	3.98	1.76	291.63

BEDDING NO. 3(B) THOROUGHLY COMPACTED SAND

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
1680	0.13	0.12	1.50	9.50	15.00	20.00	55.63	58.13	50.00	34.37	6.75	5.50	3.75	0.63	0.25	261.26
	0.19	0.37	2.62	7.50	10.88	27.19	52.82	58.13	52.82	27.19	10.88	7.50	2.62	0.37	0.19	261.27
	0.10	0.20	1.40	4.02	5.83	14.57	28.30	31.15	28.30	14.57	5.83	4.02	1.40	0.20	0.10	139.99
2500	1.13	1.13	3.88	18.13	26.25	34.37	74.37	68.75	62.50	51.88	12.50	10.50	7.75	1.50	0.88	375.52
	1.00	1.31	5.82	14.31	19.37	43.13	68.44	68.75	68.44	43.13	19.37	14.31	5.82	1.31	1.00	375.51
	0.55	0.73	3.23	7.94	10.75	23.93	37.97	38.14	37.97	23.93	10.75	7.94	3.23	0.73	0.55	208.34
3500	2.75	3.63	9.00	35.00	45.00	54.37	93.75	80.00	78.12	78.75	25.00	20.63	13.75	4.13	2.50	546.38
	2.62	3.88	11.37	27.81	35.00	66.56	85.93	80.00	85.93	66.56	35.00	27.81	11.37	3.88	2.62	546.34
	1.40	2.07	6.07	14.85	18.68	35.53	45.87	42.71	45.87	35.53	18.68	14.85	6.07	2.07	1.40	291.65

SAND CONTAINING 5.0 PER CENT MOISTURE

BEDDING NO. 1(C) LOOSE SAND

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
1680	6.00	9.75	11.62	15.00	23.75	25.62	26.25	24.38	26.25	20.00	24.37	18.75	11.25	9.75	7.75	260.49
	6.88	9.75	11.44	16.88	24.06	22.81	26.25	24.38	26.25	22.81	24.06	16.88	11.44	9.75	6.88	260.52
	3.70	5.24	6.15	9.07	12.93	12.26	14.11	13.10	14.11	12.26	12.93	9.07	6.15	5.24	3.70	140.02
2500	9.75	14.37	18.13	25.00	37.50	38.13	38.75	35.00	38.13	28.13	33.75	26.88	18.13	13.13	10.00	384.78
	9.87	13.75	18.13	25.94	35.63	33.13	38.44	35.00	38.44	33.13	35.63	25.94	18.13	13.75	9.87	384.78
	5.34	7.44	9.82	14.04	19.29	17.94	20.81	18.95	20.81	17.94	19.29	14.04	9.82	7.44	5.34	208.31
3500	11.25	20.00	26.25	35.63	55.00	53.13	55.00	47.50	51.25	40.00	50.00	36.88	25.63	17.50	13.13	538.15
	12.19	18.75	25.94	36.25	52.50	46.57	53.13	47.50	53.13	46.57	52.50	36.25	25.94	18.75	12.19	538.16
	6.61	10.16	14.06	19.65	28.45	25.24	28.79	25.74	28.79	25.24	28.45	19.65	14.06	10.16	6.61	291.66

BEDDING NO. 2(C) SLIGHTLY COMPACTED SAND

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
1680	0.63	1.00	3.25	10.00	16.25	30.00	32.50	35.63	34.37	25.00	23.13	23.12	10.63	2.37	0.62	248.50
	0.63	1.69	6.94	16.56	19.69	27.50	33.44	35.63	33.44	27.50	19.69	16.56	6.94	1.69	0.63	248.53
	0.35	0.95	3.91	9.33	11.09	15.49	18.84	20.07	18.84	15.49	11.09	9.33	3.91	0.95	0.35	139.99
2500	1.50	3.25	7.00	16.25	27.50	45.63	45.00	45.63	46.88	44.37	35.00	33.13	15.00	4.00	1.00	371.14
	1.25	3.63	11.00	24.69	31.25	45.00	45.94	45.63	45.94	45.00	31.25	24.69	11.00	3.63	1.25	371.15
	0.70	2.04	6.17	13.86	17.54	25.26	25.79	25.61	25.79	25.26	17.54	13.86	6.17	2.04	0.70	208.33
3500	3.50	7.13	12.50	30.63	45.63	64.37	60.63	60.00	61.88	59.37	46.88	46.25	23.75	7.00	2.37	531.89
	2.94	7.06	18.13	38.44	46.25	61.87	61.25	60.00	61.25	61.87	46.25	38.44	18.13	7.06	2.94	531.88
	1.61	3.87	9.94	21.08	25.36	33.93	33.59	32.90	33.59	33.93	25.36	21.08	9.94	3.87	1.61	291.66

TABLE I.—DISTRIBUTION OF PRESSURE IN THE LOWER SAND BEARING OF A 22-IN. TILE UNDER LOADS OF 1680, 2500 AND 3500 LB. PER LINEAR FOOT (*Continued*).

SAND CONTAINING 5.0 PER CENT MOISTURE (*Continued*)

Load on Tile, lb. per linear ft.	Strips as Numbered															
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
BEDDING NO. 3(C) THOROUGHLY COMPACTED SAND																
1680	0.62	2.00	5.25	10.63	15.63	34.37	38.37	42.50	35.00	30.00	20.63	11.37	5.75	1.37	1.13	254.62
	0.88	1.69	5.50	11.00	18.13	32.18	36.68	42.50	36.68	32.18	18.13	11.00	5.50	1.69	0.88	254.62
	0.48	0.93	3.02	6.05	9.97	17.69	20.17	23.37	20.17	17.69	9.97	6.05	3.02	0.93	0.48	139.99
2500	1.75	4.50	8.87	17.50	26.88	51.88	55.00	60.00	51.25	42.50	32.50	18.75	9.00	2.50	1.62	384.50
	1.68	3.50	8.93	18.13	29.69	47.19	53.13	60.00	53.13	47.19	29.69	18.13	8.93	3.50	1.68	384.50
	0.91	1.90	4.84	9.82	16.09	25.57	28.79	32.51	28.79	25.57	16.09	9.82	4.84	1.90	0.91	208.35
3500	3.25	8.25	15.63	34.37	40.62	70.63	73.12	76.88	68.12	56.88	45.00	29.37	13.75	4.25	2.00	542.12
	2.63	6.25	14.69	31.87	42.81	63.75	70.62	76.88	70.62	63.75	42.81	31.87	14.69	6.25	2.63	542.12
	1.41	3.36	7.90	17.15	23.03	34.30	37.99	41.36	37.99	34.30	23.03	17.15	7.90	3.36	1.41	291.64

SAND CONTAINING 10.0 PER CENT MOISTURE

BEDDING NO. 1(D) LOOSE SAND																
1680	5.37	7.00	10.62	15.00	21.25	26.88	21.88	26.88	22.88	23.12	23.75	18.75	13.75	9.25	6.50	252.88
	5.94	8.12	12.19	16.88	22.50	25.00	22.38	26.88	22.38	25.00	22.50	16.88	12.19	8.12	5.94	252.90
	3.29	4.50	6.75	9.34	12.46	13.84	12.39	14.88	12.39	13.84	12.46	9.34	6.75	4.50	3.29	140.02
2500	8.00	10.00	17.50	22.13	31.25	40.00	31.88	40.62	32.50	33.75	36.25	28.75	21.25	13.75	10.00	377.63
	9.00	11.88	19.37	25.44	33.75	36.88	32.19	40.62	32.19	36.88	33.75	25.44	14.37	11.88	9.00	377.64
	4.97	6.55	10.69	14.03	18.62	20.35	17.76	22.41	17.76	20.35	18.62	14.03	10.69	6.55	4.97	208.35
3500	10.00	16.25	26.25	35.00	45.00	55.63	44.37	56.25	44.37	48.13	50.62	40.00	30.00	18.75	13.75	534.37
	11.88	17.50	28.12	37.50	47.81	51.88	44.37	56.25	44.37	51.88	47.81	37.50	28.12	17.50	11.88	534.37
	6.48	9.55	15.35	20.47	26.10	28.30	24.22	30.70	24.22	28.30	26.10	20.47	15.35	9.55	6.48	291.64
BEDDING NO. 2(D) SLIGHTLY COMPACTED SAND																
1680	2.50	3.37	4.63	9.75	29.37	23.12	43.13	41.75	43.12	33.13	12.50	9.13	4.12	0.50	0.12	260.24
	1.31	1.94	4.37	9.44	20.93	28.12	43.13	41.75	43.13	28.12	20.93	9.44	4.37	1.94	1.31	260.23
	0.70	1.04	2.35	5.08	11.26	15.13	23.20	22.46	23.20	15.13	11.26	5.08	2.35	1.04	0.70	139.98
2500	4.00	6.25	7.88	14.37	45.00	34.37	56.88	51.88	58.75	51.88	24.37	17.50	10.50	2.13	0.62	386.38
	2.31	4.19	9.19	15.94	34.69	43.13	57.81	51.88	57.81	43.13	34.69	15.94	9.19	4.19	2.31	386.40
	1.25	2.26	4.95	8.59	18.70	23.25	31.17	27.97	31.17	23.25	18.70	8.59	4.95	2.26	1.25	208.31
3500	7.00	11.25	13.75	26.25	63.13	45.00	67.50	61.88	72.50	72.50	45.00	33.13	21.25	5.50	2.00	547.64
	4.50	8.37	17.50	29.69	54.07	58.75	70.00	61.88	70.00	58.75	54.07	29.69	17.50	8.37	4.50	547.64
	2.40	4.46	9.32	15.81	28.80	31.29	37.28	32.96	37.28	31.29	28.80	15.81	9.32	4.46	2.40	291.68
BEDDING NO. 3(D) THOROUGHLY COMPACTED SAND																
1680	0.50	1.75	5.25	17.50	22.50	25.00	39.37	42.50	45.00	21.25	15.00	17.50	8.00	1.37	0.63	263.12
	0.56	1.56	6.62	17.50	18.75	23.13	42.18	42.50	42.18	23.13	18.75	17.50	6.62	1.56	0.56	263.10
	0.30	0.83	3.52	9.31	9.98	12.31	22.45	22.61	22.45	12.31	9.98	9.31	3.52	0.83	0.30	140.01
2500	1.62	3.25	8.13	30.00	33.75	39.37	55.00	57.50	63.75	35.63	24.37	26.25	11.25	2.25	1.37	393.49
	1.49	2.75	9.69	28.13	29.06	37.50	59.37	57.50	59.37	37.50	29.06	28.13	9.69	2.75	1.49	393.48
	0.79	1.46	5.13	14.89	15.39	19.85	31.43	30.44	31.43	19.85	15.39	14.89	5.13	1.46	0.79	208.32
3500	3.25	5.50	13.75	43.75	50.00	59.37	69.37	71.25	78.75	55.00	38.75	38.75	17.50	4.12	2.50	551.61
	2.87	4.81	15.62	41.25	44.37	57.19	74.06	71.25	74.06	57.19	44.37	41.25	15.62	4.81	2.87	551.59
	1.52	2.54	8.26	21.81	23.46	30.24	39.16	37.67	39.16	30.24	23.46	21.81	8.26	2.54	1.52	291.65

it will be said that with bedding No. 1 the sand was shoveled loosely into the box and the tile lowered into this uncompacted sand and the load applied. With bedding No. 2 the tile was rolled 15 times back and forth across the sand so as to compact it about as ordinarily might be expected in a series of tests where no effort was made to loosen the sand of the bedding between tests of individual tile. With bedding No. 3 the tile was rolled 40 times back and forth across the sand so as thoroughly to compact the sand. This is a condition very probably approached under actual testing when no effort is made to keep the sand loose in the lower bearing. With all three beddings care was exercised that one-fourth the circumference of the tile was

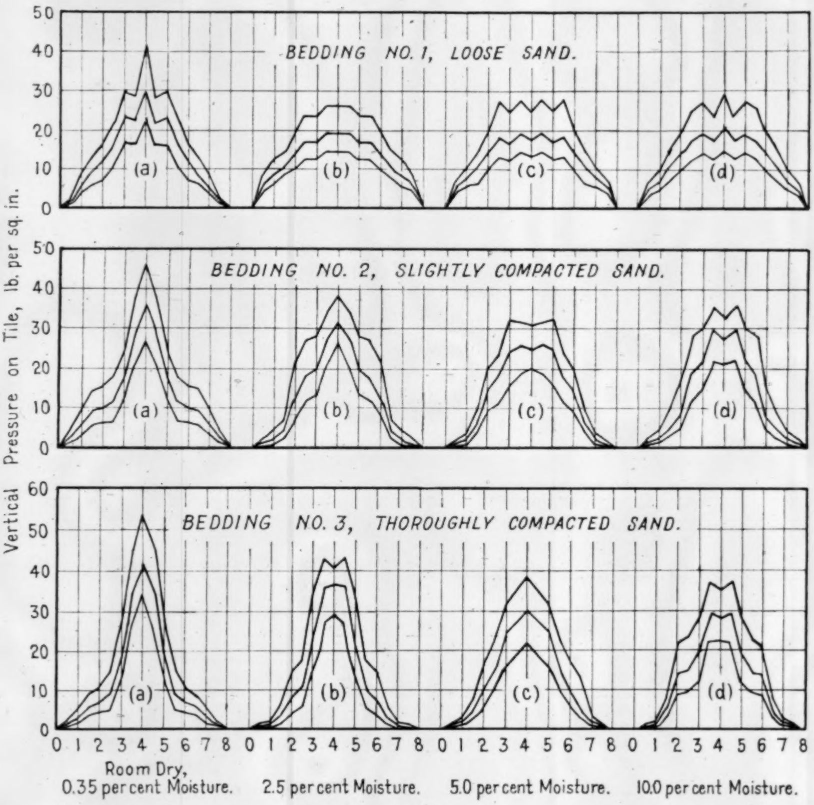

FIG. 3.—Effect of Variation of Moisture Content of Sand and Compactness Upon the Distribution of Pressure in the Lower Sand Bearing of a 22-in. Tile Under Loads of 1680, 2500 and 3500 lb. per linear foot.

bedded in accordance with the A.S.T.M. standard specifications and to this end the disturbed sand was lightly tamped back under the tile, after rolling as described in connection with beddings Nos. 2 and 3.

The tests were started with the sand room dry with an actual moisture content of 0.35 per cent as calculated by oven drying at a temperature of 240° F. Other tests were made with the moisture content increased to 2.5, 5.0 and 10.0 per cent. The tests therefore have covered three distinct bedding conditions while the sand of each bedding has had a range of 4 moisture conditions.

TABLE II.—DISTRIBUTION OF PRESSURE IN THE UPPER SAND BEARING OF A 22-IN. TILE UNDER LOADS OF 1680, 2500 AND 3500 LB. PER LINEAR FOOT.

Under each loading the first line is the average pull of two independent sets of readings, the second line is the average of corresponding readings on the two sides of the center strip, while the data in the third line are those of line 2 reduced to loads on sections, corresponding to respective strips, per linear inch of tile. Each section, therefore, measures 1 by 1$\frac{1}{16}$ in.

Load on Tile, lb. per linear ft.	Strips as Numbered														
	½	1	1½	2	2½	3	3½	4	4½	5	5½	6	6½	7	7½	Totals
ROOM DRY SAND (0.35 PER CENT MOISTURE)																
1680	10.63	10.00	11.25	12.50	20.00	21.88	21.88	29.38	23.75	21.88	15.00	10.63	10.00	10.00	11.87	240.65
	11.25	10.00	10.62	11.56	17.50	21.88	22.82	29.38	22.82	21.88	17.50	11.56	10.62	10.00	11.25	240.64
	6.55	5.82	6.18	6.73	10.18	12.73	13.28	17.09	13.28	12.73	10.18	6.73	6.19	5.82	6.55	140.03
2500	15.63	13.75	15.63	20.00	30.62	33.13	31.88	47.50	38.12	34.38	22.50	16.25	14.37	13.13	15.63	362.52
	15.63	13.44	15.00	18.12	26.56	33.75	35.00	47.50	35.00	33.75	26.56	18.12	15.00	13.44	15.63	362.50
	8.98	7.72	8.62	10.41	15.26	19.40	20.11	27.30	20.11	19.40	15.26	10.41	8.62	7.72	8.98	208.30
3500	20.00	18.75	20.00	26.88	42.50	46.88	46.88	66.87	55.63	47.50	31.87	23.13	16.88	15.63	19.37	498.77
	19.68	17.19	18.44	25.00	37.18	47.19	51.25	66.87	51.25	47.19	37.18	25.00	18.44	17.19	19.68	498.73
	11.51	10.05	10.78	14.62	21.74	27.60	29.97	39.11	29.97	27.60	21.74	14.62	10.78	10.05	11.51	291.65
SAND CONTAINING 5.0 PER CENT MOISTURE																
1680	16.25	8.75	9.37	13.13	16.88	20.62	24.37	26.25	24.37	21.25	15.63	10.00	8.75	10.63	13.37	239.62
	14.81	9.69	9.06	11.56	16.25	20.94	24.37	26.25	24.37	20.94	16.25	11.56	9.06	9.69	14.81	239.61
	8.65	5.66	5.29	6.75	9.49	12.23	14.24	15.34	14.24	12.23	9.49	6.75	5.29	5.66	8.65	139.96
2500	22.50	11.68	11.68	20.62	28.13	32.50	35.63	40.63	37.50	32.50	21.25	18.75	12.50	15.00	20.00	360.87
	21.25	13.34	12.09	19.68	24.69	32.50	36.56	40.63	36.56	32.50	24.69	19.68	12.09	13.34	21.25	360.85
	12.27	7.70	6.98	11.36	14.25	18.76	21.11	23.46	21.11	18.76	14.25	11.36	6.98	7.70	12.27	208.32
3500	26.25	15.63	15.63	30.00	38.75	48.13	50.00	59.37	53.13	49.37	30.63	26.88	16.88	17.50	25.00	503.15
	25.62	16.57	16.25	28.44	34.69	48.75	51.56	59.37	51.56	48.75	34.69	28.44	16.25	16.57	25.62	503.13
	14.85	9.61	9.42	16.49	20.11	28.26	29.89	34.42	29.89	28.26	20.11	16.49	9.42	9.61	14.85	291.68

CONCLUSIONS

Perhaps the most significant points developed by these tests are the following:

1. For each of the three beddings, dry sand is the least satisfactory as in all cases the concentration of loading has been greatest with dry sand.

2. Up to the limit of the holding capacity of the sand, in this case about 10.0 per cent, the quantity of moisture does not seem to be of very great importance although perhaps the most consistent results were obtained with a moisture content of 5 per cent.

3. Loose sand is preferable to any degree of compactness, for while with moist loose sand the pressure distribution is rather parabolic than uniform it is more nearly uniform than under the other conditions.

In general the results of the tests for the lower bearings would indicate the necessity for specifying loose sand with a moisture content of about 5 per cent. By loose sand is meant such as results when the sand in the bottom bearing is loosened by spading immediately before placing the tile for the test.

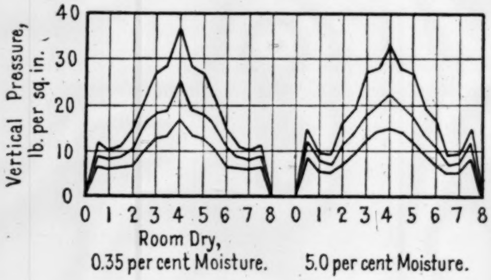

FIG. 4.—Effect of Variation of Moisture Content of Sand upon the Distribution of Pressure in the Upper Sand Bearing of a 22-in. Tile Under Loads of 1680, 2500 and 3500 lb. per linear foot.

From the nature of the test the compactness of the sand in the top bearing need not be given consideration nor does the moisture content have much influence. Tests, however, were made of the top bearing to determine whether any appreciable difference in pressure distribution resulted between room dry sand and sand with 5 per cent moisture. The data of these tests are shown in Table II from which the graphs of Fig. 4 were constructed after reducing the load to pressure in pounds per square inch. It will be noted that while in the top bearing the pressure distribution is not uniform it is slightly more so when the sand is moist so that in general 5 per cent moisture for both top and bottom bearings is perhaps about as satisfactory a condition as can be realized.

MEASUREMENT OF CONSISTENCY AS APPLIED TO RUBBER-BENZENE SOLUTIONS[1]

By Winslow H. Herschel[2] and Ronald Bulkley[3]

Synopsis

By the use of two Bingham viscometers and two Ostwald viscometers arranged to be used with external pressure, the consistency was measured of benzene solutions of unmilled crepe rubber, with concentrations of 0.2 to 1.2 per cent. These tests showed that the solutions were all plastic, that is, the rate of flow was not proportional to the pressure as with a truly viscous liquid. The consistency of a plastic material can not be expressed by a single numerical value, such as "apparent viscosity," but must be expressed by an equation or other means which completely defines the variable relation between the rate of flow and the force which produces it, such as by means of the yield shear value and mobility. However, it appears preferable to use "stiffness," the reciprocal of mobility, in order to express consistency by two factors both of which affect the rate of flow in the same direction. Even with this modification, the method is open to the objection that many points fall upon the curved portion of the flow-pressure graph and cannot be utilized. It was found that for rubber-benzene solutions, values of yield shear value and stiffness were obtained which varied with the dimensions of the capillaries used, and more concordant results were obtained by using the constants I and n of the empirical equation,

$$\frac{d^4 (P-K)^n}{q\,l} = I$$

where d and l are the diameter and length of capillary, P is the pressure and q is the rate of flow. The exponent, n, is a constant of the material which is independent of the dimensions of the capillary and increases with the concentration. I is another constant of the material which also increases with the concentration and may be tentatively assumed to be independent of the dimensions of the capillary. K is perhaps an unknown function of a third constant of the material and of the dimensions of the capillary, but it vanishes at low concentrations with large capillaries and I and n should ordinarily prove adequate to define the consistency of a plastic material to which the above equation is applicable.

Introduction

As pointed out in a preliminary paper[4] a 1-per-cent solution of crude rubber in benzene is plastic, and this has been found to hold true even for a concentration as low as 0.2 per cent. In this investigation the Ostwald viscometer, calibrated as previously described,[1] was used because considered standard for rubber solutions,[2] and the Bingham instrument[3] was used later in order to get more accurate results.

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[1] Published by permission of the Director of the Bureau of Standards, U. S. Department of Commerce.

[2] Associate Physicist, U. S. Bureau of Standards, Washington, D. C.

[3] U. S. Bureau of Standards, Washington, D. C.

[4] Winslow H. Herschel, *Industrial and Engineering Chemistry*, Vol. 16, p. 927 (1924).

Solutions were prepared from a sample of unmilled pale crepe rubber by cutting the sheet into about 50 pieces per gram, and dissolving the rubber in enough benzene to give approximately the concentration desired. The true concentration was determined by evaporating to dryness, duplicate portions of the solution weighed out at the time of filling the viscometers. Solutions were made up in glass bottles which were kept in the dark and stoppered with corks

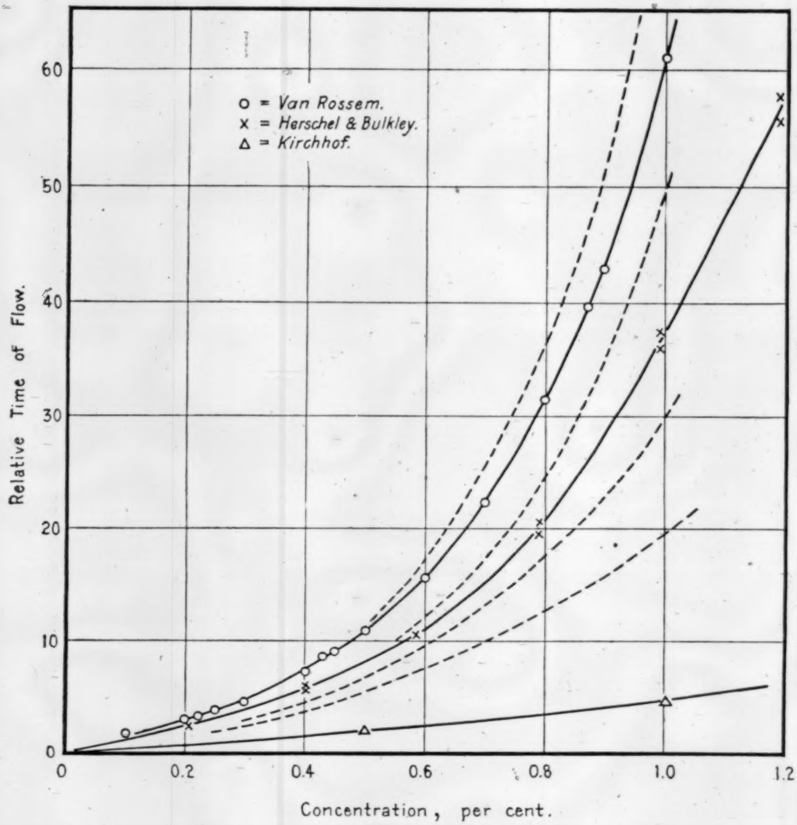

FIG. 1.—Variation in Relative Time of Flow with the Concentration.

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[1] Winslow H. Herschel and Ronald Bulkley, *Journal Physical Chemistry*, Vol. 29, p. 1217 (1925).

[2] A. van Rossem, *Koll. Chem. Beihefte*, Vol. 10, p. 83 (1918–19).

[3] E. C. Bingham, "The Variable-Pressure Method for the Measurement of Viscosity," *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part II, p. 373 (1918).

covered with tin foil. Determinations of consistency were made after the solutions had stood for 24 hours with an occasional shaking by hand. Shaking on a mechanical shaker was found to give solutions which showed a shorter time of flow. Practically all of the rubber went into solution. Filtering was done through fat-free wool, although glass wool was found to be equally satisfactory when it was placed in a layer over the bottom of a Gooch crucible and the solution was filtered without suction. Technical benzene was redistilled over sodium hydroxide before using and was found to give the same results as c. p. thiophene free benzene.

VARIATION OF APPARENT VISCOSITY WITH THE CONCENTRATION

The well-known equation for stream-line flow of viscous liquids in capillary tubes may be conveniently written

$$\mu = C\left(P_m + h_o\,\rho - \frac{B\,\rho}{C\,t^2}\right)t = C\,P\,t \ldots\ldots\ldots\ldots (1)$$

where P_m is the pressure calculated from the manometer reading, h_o is the hydrostatic head and the last term in the parenthesis is the kinetic energy correction expressed as a pressure. If the value of μ calculated from Eq. 1 is a constant, independent of the pressure used in tests, this value is the viscosity in poises. Otherwise the value obtained is the so-called "apparent viscosity." A material will be considered plastic if μ is a variable, and its consistency must be expressed by an equation or other means which completely defines the variable relation between pressure and rate of flow.

TABLE I.—DIMENSIONS AND OTHER DATA CONCERNING VISCOMETERS.

	Ostwald No. 1	Ostwald No. 2	Bingham No. 3	Bingham No. 2
d, cm	0.0811	0.0840	0.1018	0.0689
l, cm	12.3	10.3	10.0	14.6
qt, cc	2.740	2.325	3.970	3.985

The variation in apparent viscosity with the concentration of rubber solutions is shown in Fig. 1.[1] The dotted lines, reproduced from a diagram of van Rossem, show typical graphs for different grades of rubber. If the conclusion is correct that it is impossible to completely define the consistency of a plastic material by a single numerical value, then all attempts to determine the laws of variation of apparent viscosity with the concentration are futile.[2]

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[1] F. Kirchhof, *Koll. Zeit.*, Vol. 15, p. 30 (1914).

[2] E. Hatschek, *Koll. Zeit.*, Vol. 11, p. 284 (1912); G. S. Whitby, "The Theory and Application of Colloidal Behavior" (R. H. Bogue, Editor), Vol. 2, p. 652 (1924).

YIELD SHEAR VALUE AND STIFFNESS

The consistency of a plastic material is often described, approximately, by its yield shear value, f, and its mobility. It is desirable to use in place of mobility some factor which, like f, increases with increased resistance to flow, so that the phrase "more plastic" may have an intelligible meaning. The term stiffness, S, is suggested as a suitable name for the reciprocal of mobility. Hence we have available to express consistency:

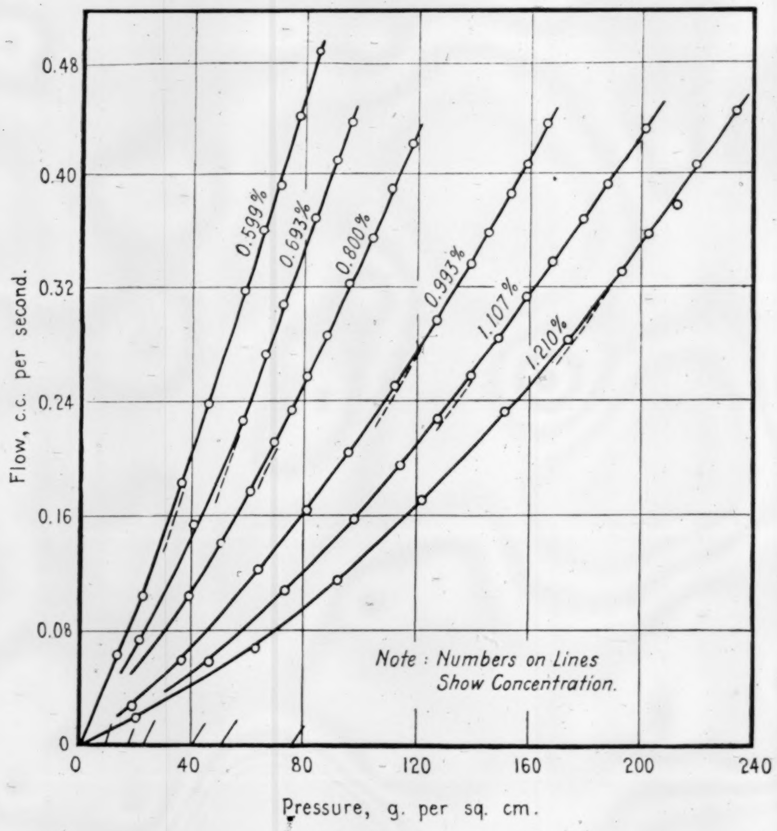

FIG. 2.—Flow-Pressure Graphs, Bingham Viscometer No. 3.

$$f = \frac{pdg}{4l} \quad \text{.........................} \quad (2)$$

$$S = \frac{\pi\, g\, d^4\, (P - p)\, t}{128\, Q\, l} = \frac{(P - p)\, C\, q\, t}{q} \quad \text{..........} \quad (3)$$

When the rate of flow q is plotted against the corrected pressure P, as given in Eq. 1, to obtain the usual flow-pressure diagram, p is obtained as the intercept of the upper straight portion of the graph, prolonged, on the axis of abscissas. Stiffness is equal to the product of $\frac{P-p}{q}$, obtained graphically, the instrumental constant C and the volume of the viscometer bulb, qt.

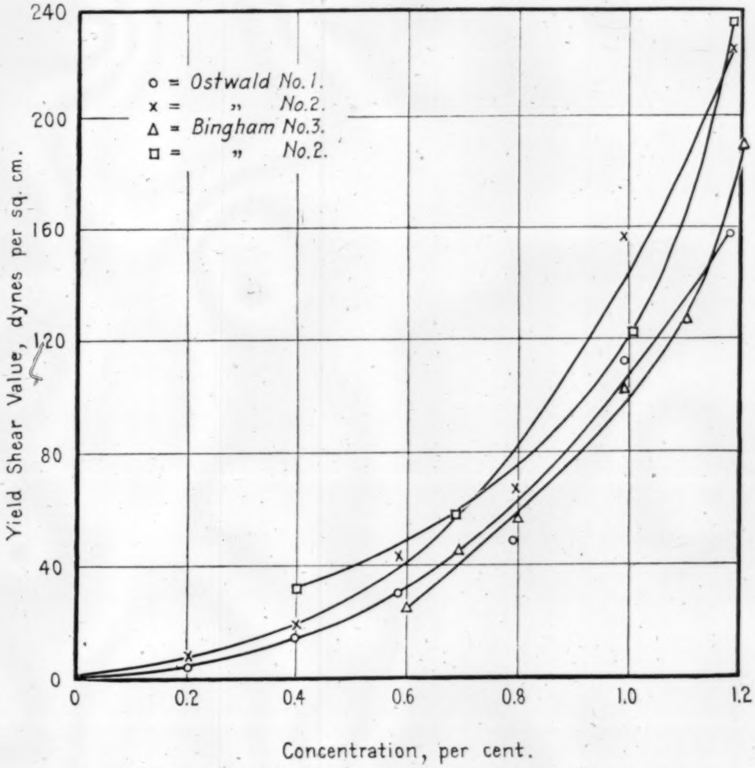

FIG. 3.—Change in Yield Shear Value with the Concentration.

It is believed that the mean effective diameter of capillary, d, for use in Eq. 2 may be obtained most accurately by means of flow tests. Values thus obtained are given in Table I.

In using the Ostwald viscometers at high pressures, it was found impossible to shut off the pressure quickly enough to avoid blowing air through the capillary, as the Ostwald instrument is not provided, as is the Bingham, with an auxiliary bulb below the measuring bulb. The Bingham viscometers were used to avoid this difficulty, results obtained with instrument No. 3 being shown on Fig. 2. As may be seen from Fig. 3, the relation between yield shear value and concentration is not linear, as found for mixtures of lithopone and linseed oil,[1] but, from Fig. 4, there is an approximately linear relation between stiffness and concentration.

THE LOGARITHMIC FLOW-PRESSURE DIAGRAM

The discrepancy in the values of yield shear value and of stiffness obtained with capillaries of different size is in accord with previous experience. In the light of present knowledge it seems quite possible that any one method or equation for expressing the consistency of a plastic material may not have universal application. Indeed, each separate class of materials may demand individual treatment.[2]

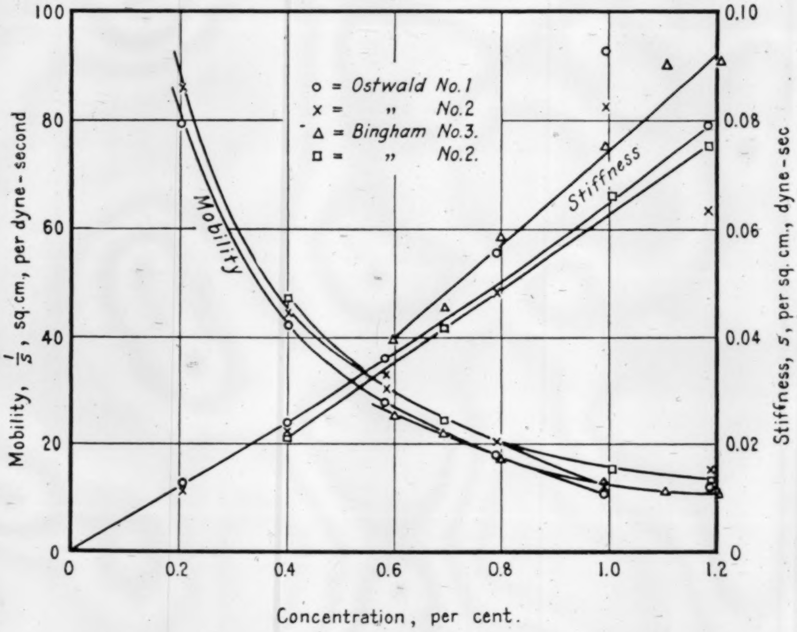

FIG. 4.—Change in Stiffness and Mobility with the Concentration.

Farrow and Lowe[3] in tests on starch pastes found that plotting the logarithm of the rate of flow against the logarithm of the pressure gave a straight line, so that:

$$q = k\,P^n \text{ or } q = k'\,(gP)^n \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

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[1] E. C. Bingham, "The Theory and Application of Colloidal Behavior" (R. H. Bogue, Editor), Vol. 1, p. 441 (1924).

[2] E. C. Bingham, Colloid Symposium Monograph, Vol. 2, p. 109 (1925).

[3] F. D. Farrow and G. M. Lowe, *Journal*, Textile Inst., Vol. 14, p. T 414 (1923); see also P. G. Nutting, "A Study of Elastic Viscous Deformation," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1162 (1921).

where n is a constant of the material, and k or k' is an unknown function of another constant of the material, I, and of the dimensions of the capillary.

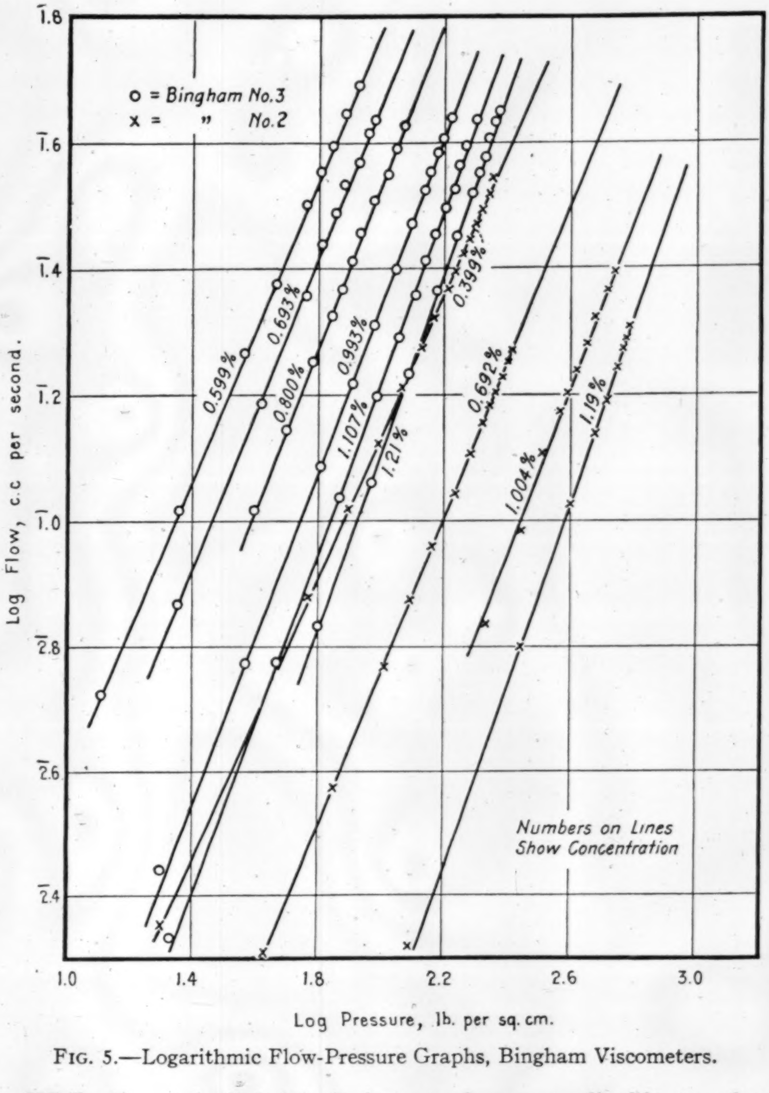

FIG. 5.—Logarithmic Flow-Pressure Graphs, Bingham Viscometers.

While Eq. 4 is helpful, it is not always applicable to plastic materials. It was pointed out by Green[1] and also by Porst and

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[1] Henry Green, "Further Development of the Plastometer and its Practical Application to Research and Routine Problems," *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part II, p. 451 (1920); Henry Green and G. S. Haslam, *Industrial and Engineering Chemistry*, Vol. 17, p. 726 (1925).

Moskowitz[1] that sometimes the flow-pressure graph does not pass through the origin. This difficulty may be overcome by writing:

$$q = k'' \, (gP - A)^n \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

where A must be found by trial so that a straight graph is obtained if the logarithm of q is plotted against the logarithm of $(gp - A)$.

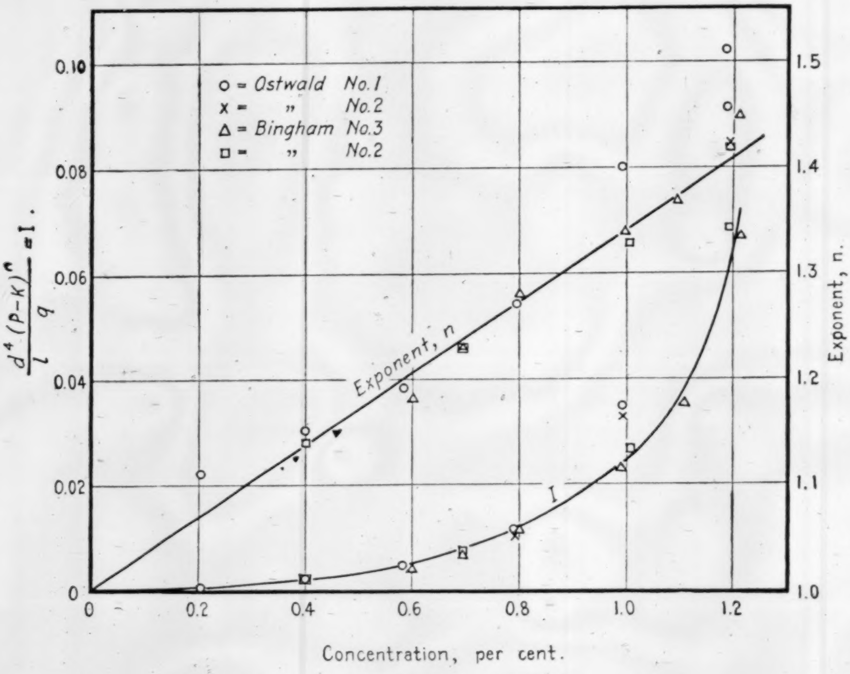

FIG. 6.—Change in n and I with the Concentration.

Taking into consideration the empirical nature of all available equations for plastic flow, it is convenient to write,

$$\frac{d^4}{l} \, \frac{(P - K)^n}{q} = I \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Equation 6 appears to be the most general equation available for plastic flow of certain colloidal solutions through capillary tubes.

Figure 5 shows logarithmic flow-pressure graphs as obtained with the Bingham instruments. For the two highest concentrations with viscometer No. 2, the abscissas are values of log $(P - K)$; for all other graphs, K was found to be negligible. Values of n and I

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[1] C. E. G. Porst and M. Moskowitz, *Industrial and Engineering Chemistry*, Vol. 15, p. 166 (1923).

are shown in Fig. 6 for all four viscometers. Considering the more accurate tests with the Bingham instruments, it will be noted that n varies almost linearly with the concentration. The better agreement between different instruments shown in Fig. 6, as compared with Figs. 3 and 4, indicates that Eq. 6 is better than Eqs. 2 and 3 as a means of expressing the laws of flow of this type of plastic material. Unless it should prove desirable to obtain three constants of the material, it might be possible and preferable, by the use of large capillaries, to render K negligible, and thus avoid the labor and uncertainty involved in its determination.

CONCLUSIONS

1. Of equations expressing the flow through a capillary tube,

$$\frac{d^4\,(P - K)^n}{l\,q} = I$$

is the best available for benzene solutions of crepe rubber from 0.2 to 1.2-per-cent concentration.

2. The method of measuring consistency by the use of this equation has the advantage of utilizing data at practically all rates of flow, and of avoiding the need of using very high pressures.

3. Avoidance of high pressures permits the use of simpler apparatus, and reduces the error due to uncertainty in regard to the kinetic energy correction. It would also make it possible to make use of the Ostwald viscometer, with moderate external pressures, without danger of blowing air through the capillary at the end of a run.

4. By the use of the logarithmic flow-pressure diagram, closer agreement can be obtained between capillaries of different dimensions than by determinations of yield shear value and stiffness (or mobility).

5. The constants of the material n and I, obtained from the logarithmic graph, both increase with the concentration. As compared with yield shear value and mobility, these constants have the advantage that they both affect the flow in the same sense, so that "more plastic" could be interpreted as indicating higher values of n and I.

6. K vanishes or is too small to be detected at low concentrations with large capillaries. If of appreciable size, its value increases as the diameter of the capillary decreases. K of itself is not a constant of the material, but further experience might indicate that a third constant can be calculated from it.

DISCUSSION

Mr. Fulweiler. THE CHAIRMAN (*President W. H. Fulweiler*[1]).—Mr. Herschel's paper is open for discussion. Is it proper for the chairman to ask what is the order of magnitude of the difference in the yield value and the intercept of the experimentally determined graph on the pressure axis?

Mr. Herschel. MR. WINSLOW H. HERSCHEL.—Ordinarily you do not get a straight graph but one that turns off towards the origin, but you do not get data down low enough to find the actual point where the graph intersects the pressure axis.

Mr. Fulweiler. THE CHAIRMAN.—That was just the point I wanted to bring out.

Messrs. Herschel and Bulkley. MESSRS. WINSLOW H. HERSCHEL AND RONALD BULKLEY (*authors' closure by letter*).—The chairman's question may be answered more completely by reference to the accompanying Fig. 1. According to Bingham and Green,[2] a straight graph passing through the origin on Fig. 1(*a*) would represent a viscous liquid, but any other form of graph would indicate a plastic material. Their idea appears to have been that *BF* is the true graph for a plastic material, although on account of slippage or seepage, *BD* is actually obtained. Hence they regarded *OF* as the yield value, although *F* can not be obtained experimentally and must be found by prolonging *BC*, the straight or approximately straight upper portion of the graph *BD*. According to the report of a sub-committee of Committee E-1 on Methods of Testing,[3] "the yield value is commonly taken to represent the load which can be applied to a plastic body without causing any deformation." It is not clear whether this defines *OE* or *OF*, but in what follows, *OE* will be called the "starting pressure" as proposed by Porst and Moskowitz, and *OF* will be called the yield value. Letting $OE/OF=R$, the difficulties in obtaining accurate values of *R* will be considered.

It is difficult to locate *E* because it requires extrapolation on a curve. If the flow-pressure graph takes the form *GH*, in which the approximately straight upper and lower sections are connected by

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[1] Chemical Engineer, United Gas Improvement Co., Philadelphia, Pa.

[2] E. C. Bingham and Henry Green, "Paint, a Plastic Material and not a Viscous Liquid; the Measurement of Its Mobility and Yield Value," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, p. 640 (1919).

[3] Report of Sub-Committee on Consistency, Plasticity, etc., *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 516 (1926).

Messrs. Herschel and Bulkley.

a curve, the determination of the starting pressure may be equally difficult on account of the small angle between the graph and the pressure axis. Graphs both of the types of *BD* and *GH* are given by Hall[1] for clays and by Porter and Gruse[2] for worked cup greases, and in many cases it is impossible to estimate whether or not the graph

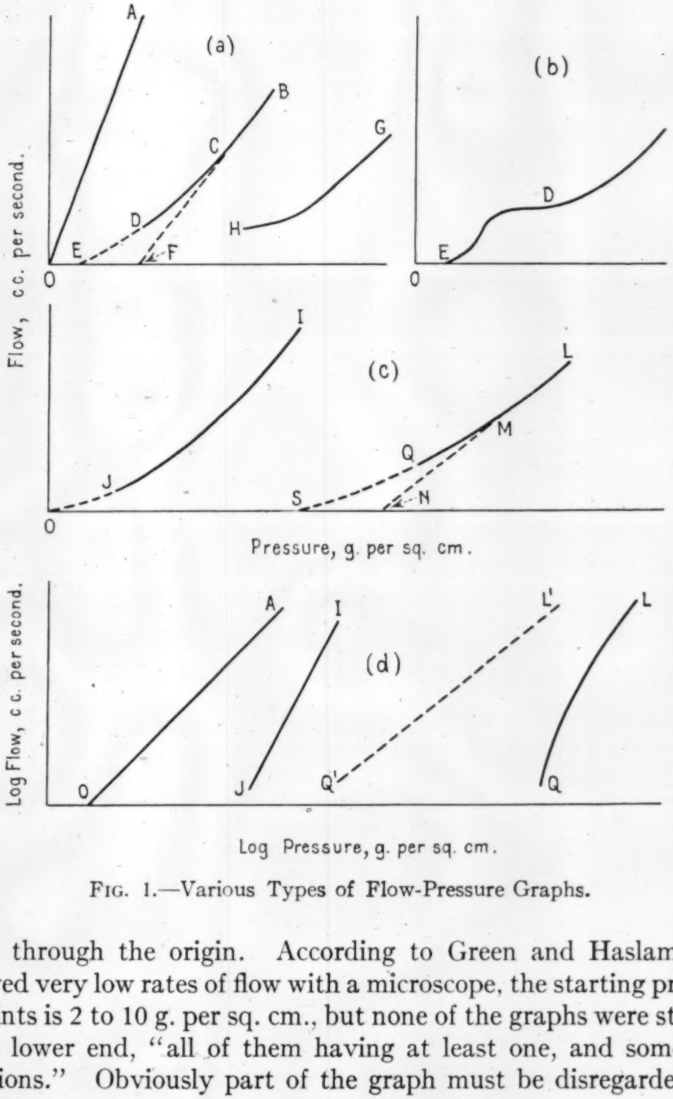

FIG. 1.—Various Types of Flow-Pressure Graphs.

passes through the origin. According to Green and Haslam, who observed very low rates of flow with a microscope, the starting pressure for paints is 2 to 10 g. per sq. cm., but none of the graphs were straight at the lower end, "all of them having at least one, and some two, inflections." Obviously part of the graph must be disregarded if it

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[1] F. P. Hall, U. S. Bureau of Standards *Technologic Paper No. 234* (1923).

[2] P. K. Porter and W. A. Gruse, *Industrial and Engineering Chemistry*, Vol. 17, p. 953 (1925).

Messrs. Herschel and Bulkley.

takes the form shown in Fig. 1(*b*), and *E* can not be obtained by extrapolation.

If, as frequently happens, *BC* is located by an inadequate number of points, and is short in comparison with *CF*, even the point *F* can not be accurately located. However, locating *E* and *F* as accurately as possible from the data available, it may be said that *R* seldom exceeds 0.5.

There is less difficulty in determining *R* when the flow-pressure graphs are of the form shown at *LQ*, Fig. 1(*c*). There is still the difficulty that *MN* may be long in comparison with *LM*, and that *LQ*, prolonged, makes a poor intersection with the pressure axis. The point *S*, however, may be located by the help of the logarithmic flow-pressure diagram, Fig. 1(*d*), on which *AO*, with a 45-deg. slope, represents a viscous liquid, and *IJ* is the graph of a plastic material whose consistency is defined by Eq. 4 of the paper. Since the exponent n is greater than unity, *IJ* will have a steeper slope than *AO*. Finally, *LQ* represents a plastic material in agreement with Eq. 6, and *L′Q′* is the straight graph obtained by plotting log q against log $(P\text{-}K)$. Now K is the starting pressure, or value of *OS* in Fig. 1(*c*), and for the two cases in tests of rubber-benzene solutions where the graph did not pass through the origin, K was found to be 10 and 15 g. per sq. cm. Using these values, and finding *ON* by graphical extrapolation, *R* was found to be 0.10 and 0.07 for the 1.004 and 1.19-per-cent solutions respectively. In all other cases with rubber-benzene solutions, *OS* and hence *R* was zero or negligible.

At the other extreme, if a flow-pressure graph shows no curvature, as in the tests of Bingham and Murray,[1] *R* is equal to unity, but this value appears to require confirmation, as the yield value obtained, 18.1 dynes per sq. cm., was not in accord with values of 110 and 106 previously obtained for the same material. With this exception the highest value of *R*, 0.61, appears to have been obtained by Porst and Moskowitz for starch pastes, though most of the graphs show values of less than 0.50. They say, "By the usual practice the pressure is gradually raised from the point where there is no flow until flow just occurs. It has been found that the pressure determined by logarithmic plotting (the pressure at which flow starts) is close to the pressure found by experiments."

It should be noted that Eq. 6 has only been applied to rubber-benzene solutions and to starch pastes, and it remains to be determined to how many types of material it is applicable. At present

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[1] E. C. Bingham and H. A. Murray, Jr., "A New Combined Viscometer and Plastometer," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 655 (1923).

Messrs. Herschel and Bulkley.

it appears reasonable to expect that this or some similar equation is typical of emulsoids, while suspensions are apparently better represented by the graphs *BD* and *GH* of Fig. 1(*a*), which are practically straight at the upper ends. Although the method of defining consistency of suspensions by Eqs. 2 and 3 is not entirely satisfactory, on account of the uncertainties in determining p, and the observed discordance between values of f determined with capillaries of different dimensions, we are not prepared to suggest any better method.

IMPACT TESTING OF INSULATING MATERIALS

By W. W. Werring[1]

Synopsis

This paper is a discussion of the application of the impact test to the insulating materials and of the development of low capacity pendulum type machines of sufficient sensitivity for the purpose.

Both Charpy and Izod type tests are discussed and comparative data shown. Results of static flexure tests of both plain and notched bars are given in comparison with similar impact test results. The effect of initial energy of impact and initial velocity of impact are shown by graphs.

The conclusions drawn are (*a*) that insulating materials possess impact properties not indicated by static tests even of notched bars, (*b*) that a comparative test of either Charpy or Izod type will probably be satisfactory in this field, (*c*) that great weight of machine is not essential in the capacities required, (*d*) that the magnitude of the initial energy of impact is of little significance to the results, except as it involves the sensitivity of the machine, (*e*) that the velocity of impact is of great effect and that standardization of velocity is essential.

This paper covers a preliminary investigation of the impact testing of insulating materials made by the Bell Telephone Laboratories in connection with its general study of the physical properties of molded insulation. Being carried on concurrently with similar work initiated earlier by the Society's Committee D-9 on Electrical Insulating Materials, upon which the laboratories are represented, this investigation was prosecuted with the helpful cooperation of the other interested members of the committee. The experience of the committee in the development and investigation of suitable testing apparatus was of considerable assistance and some data are quoted from the work of the members.

The materials considered are probably, as a class, more brittle than those previously investigated in impact and the samples in a given lot do not appear so subject to wide variations from minor differences in past history as do the metals. It is felt that the information obtained from several hundred tests on a variety of such materials will be of considerable interest even outside the special field of the insulating materials.

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[1] Bell Telephone Laboratories, Incorporated, New York City.

IMPACT TEST AS APPLIED TO METALS

The notched-bar test as applied to metals has probably found its greatest usefulness as an arbitrary, strictly comparative test, and has admittedly established itself as a means of determining for a given material its maximum properties for resistance to suddenly applied localized stresses. A variety of machines and types of notch and test specimen have been used, and certain features of the test have been more or less arbitrarily standardized by various foreign authorities. Such standardization, though urged in the United States, does not really satisfy the need for an impact test of general application. The present test distinguishes materials which are unsuited to use under impact loads from those which are better suited but does not give quantitative data directly useful in design. If materials have impact properties which differ from static properties, the ultimate aim of impact testing must be to develop a test which will yield values of impact strength in terms of unit dimensions and method of load application and the other factors which enter into the design of structures and mechanisms. That the impact test of metals does not now afford data of this kind is a major objection to it and there seems little likelihood that this objection can readily be overcome, as the test is intentionally made one of behavior under relatively high localized stress concentrated in the region of the bottom of the notch and therefore cannot be readily related to the dimensions of the specimen. The safest course appears to be to state the strength only in terms of a standard test piece, but it is only necessary to consider the situation that would exist were tension test results so stated to realize the limitations that this alone imposes.

Another factor in the problem is the velocity with which the specimen is struck. That at least certain of the metals show widely different impact figures when tested at different velocities of impact is now rather generally accepted. In present practice, tests are made at a variety of speeds depending upon the machine used, and the use of the results as anything but strictly comparative data is impossible.

The widespread ramifications of even these factors are beyond the scope of this paper but even this brief mention of them will indicate how remote is the possibility of putting the notched-bar impact test of metals on a rational basis so as to give data applicable to problems of design. That this is so does not necessarily reduce the value of the test as a strictly comparative one, but it does indicate its limitations and the causes of the objections to its standardization as a general test.

APPLICATION OF IMPACT TESTS TO INSULATING MATERIALS

In the case of the impact test for insulating materials the problem is much simplified. These materials, in general, are not called upon to act primarily as load-carrying members and are not usually designed on the basis of strength calculations, as the electrical requirements and space limitations often have much to do with the choice of material and the shape and even the size of the part. These materials, however, are in many cases subjected to severe accidental impact in installation or service and resistance to such impact, while a minor factor in design, may be the determining one in length of service life. In such cases, a test is needed for use in the development of a given material to its maximum resistance to impact, a field in which the strictly comparative test is quite sufficient.

The mouthpiece and the receiver shell or case of the telephone illustrate the general conditions of service outlined and the application of the impact test to such materials. Obviously, when not abused the mechanical requirements of service for these parts are not severe, but with lamentable frequency the average desk stand telephone is crowded off the desk to crash to the floor often to the serious detriment of the mouthpiece. In other cases, the stand is caught as it topples over and the receiver swings at the end of its cord to pound heavily against the desk. The shape and size of both these parts are now rather rigidly fixed and strength is almost entirely a problem of material, including material cost. It is quite an important problem as the materials' resistance to blows determines to a large extent the frequency of replacement and the effect of any changes in compounding or manufacture on the impact strength of these parts must therefore be carefully determined in the laboratory. This can be done by comparative full scale tests of parts of the different materials simulating as closely as possible the treatment encountered in service, but while accurate when carefully carried out, such tests require considerable skill and judgment. The need for a simple means of evaluating the impact strength of the materials used for such purposes has been one long recognized, but until recently no such means were available.

A contemplated change in the process of manufacture of the receiver shell afforded an excellent opportunity for a practical trial of the newly developed pendulum type machines as applied to this typical problem. Test results on suitable specimens milled directly from the different lots of receiver shells gave results not only parallel to those obtained by the full scale service test but even agreeing rather closely in the actual percentage differences.

Another promising application of the test to process control work is in the case of the phenolic-resin molded products which require a certain period of curing in the die to attain their final infusible condition. The length of this cure is very largely a matter of judgment based on the size of the part and the type of die. At present this estimate is supplemented by the "acetone test" in which the finished part is placed in acetone for 15 minutes and considered cured if not affected. Limited tests made on lots of a conveniently shaped part which were cured for 1, 2, 3, 4 and 5-minute periods showed that, whereas the acetone test indicated all but the 1-minute cure to be sufficient, the impact test on the whole parts showed an increase in strength from a minimum with the 1-minute cure to a maximum with the 4-minute cure with a decrease from this maximum in the parts cured 5 minutes. These effects were accompanied by a slight increase in both flexure strength and deflection at break with cures from 3 to 5 minutes. Curiously enough, the insulation resistance seemed to show a somewhat similar maximum with the 4-minute cure for the particular part and material tested.

These are but preliminary indications of the possibilities of the test. They are of particular interest in that they give evidence of two quite important points. In the case of the receiver shell, impact tests of small unnotched specimens cut from the finished product gave the same results as tests of the whole part, and in the case of the molded phenol plastics the decrease in impact strength of parts cured 5 minutes without an accompanying decrease of either flexural strength or deflection indicated an apparent difference in impact and static properties.

DEVELOPMENT OF LOW-CAPACITY IMPACT MACHINES

The special testing machine mentioned as used in these tests was a development made necessary by the fact that the standard machines in use in the testing of metals are of high capacity and lack the sensitivity required for the testing of the extremely weak and brittle molded materials. Values of impact strength as low as 0.15 ft-lb. are not infrequently encountered with a $\frac{1}{2}$-in. specimen and it is obvious that a machine striking a blow of 100 ft-lb. or even of 25 ft-lb. could not be expected to indicate such a minute absorption of energy with any degree of accuracy. This lack of a suitable impact testing machine was recognized as a considerable handicap to those interested in such materials and Committee D-9 early turned to the development of a machine of a capacity suited to this work.

Ohio Brass Co. Machine:

As a result, Mr. F. L. Wolf of the Ohio Brass Co. designed and built a machine of about 7 ft-lb. capacity intended to be used for the testing of light alloys as well as of insulating materials. It was planned to be convertible so as to make either the Izod or the Charpy test but was originally built as the Izod as shown in Fig. 1. The machine is constructed of several substantial castings forming the base and bearing supports for a pendulum arm about 30 in. long. It is arranged to release the pendulum from any position up to a maximum inclination of 60 deg. The pendulum swings in carefully aligned plain bearings and carries with it a pointer which is held by a friction device at the highest position reached after breaking the specimen, indicating on a scale the angle of swing from which the residual energy may be computed.

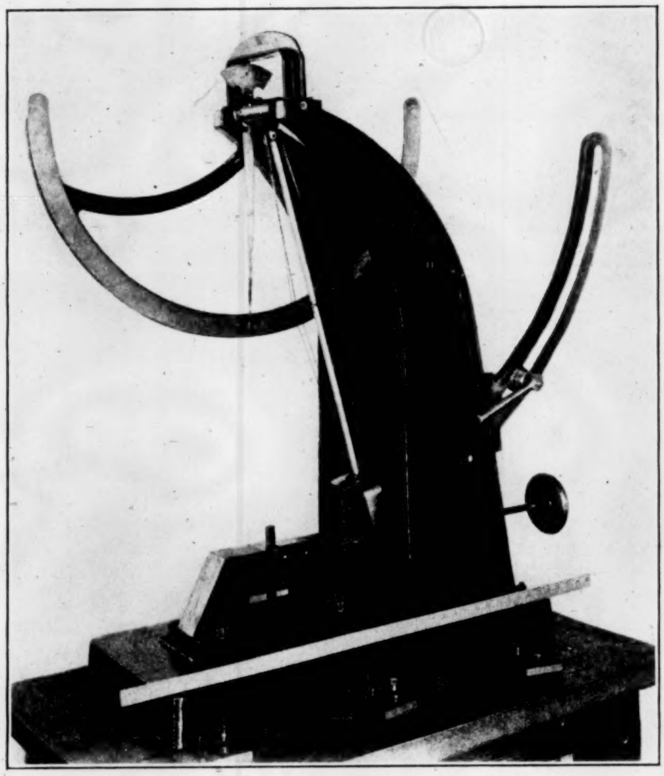

FIG. 1.—Izod Type Machine Developed by Ohio Brass Co.

Photograph by courtesy of the Bakelite Corporation.

Later, Mr. T. S. Taylor, of the Bakelite Corporation, and Mr. M. P. Davis, of the Otis Elevator Co., both built and experimented with duplicates of this machine. It was found necessary to redesign the original pointer arrangement to reduce the friction losses and Taylor modified his machine in this particular and, it is understood, used it with considerable satisfaction. The impact values of many of the materials tested were found to be so low as to require that the pendulum be dropped from a very low height and with a correspondingly low velocity in order to increase the sensitivity of the machine.

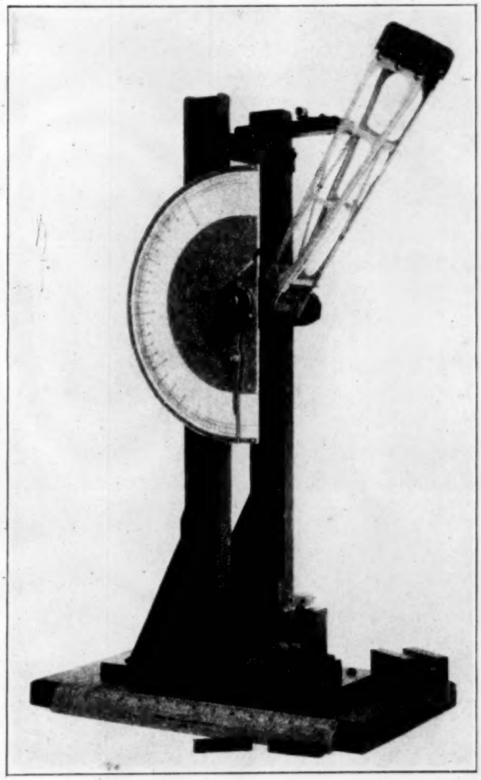

FIG. 2.—Small Izod Type Machine Developed by the Bell Telephone Laboratories.

Bell Telephone Laboratories Machine:

Some time after this, the author built at the Bell Telephone Laboratories two small light-weight machines of very low capacity intended only for use on the insulating materials. One is a Charpy type of 3 ft-lb. capacity, the other an Izod of capacity variable from 2 ft-lb. to 5 ft-lb. It was felt that a fairly high velocity of impact was desirable and that all tests should be made at the same velocity so the machines were originally built with a fixed height of drop of 2 ft. corresponding to a striking velocity of over 11 ft. per sec. which is of the order of the velocity of the larger machines used for metals. In order to meet the requirements for both sensitivity and capacity, the pendulum was built to an effective weight of only 1.5 lb. for the Charpy and 1 lb. for the Izod but arranged in the case of the latter for the addition of calibrated weights so attached as to raise the capacity, in steps, to 5 ft-lb. without affecting the location of the center of percussion of the hammer or other characteristics of the machine. In order to fulfill the weight and size requirements without sacrificing rigidity, the pendulum arm was made of truss construction cut from

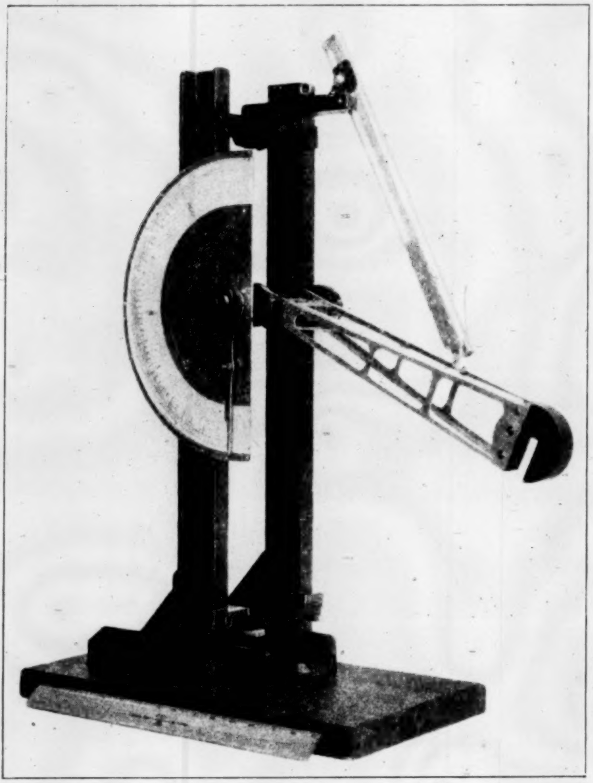

FIG. 3.—Small Charpy Type Machine Developed by the Bell Telephone Laboratories.

sheet aluminum and only 13 in. from center of suspension to the center of percussion which is coincident with the striking edge. The construction of these machines is clearly shown by Figs. 2 and 3. In operation the pendulum swings through an angle of about 135 deg. as shown in Fig. 2 giving a rather open scale. As can be seen, both machines are simply constructed and are of small size, the base being 10 by 18 in. and the height of the uprights about 26 in. The Izod machine is adapted to be quickly converted to the Charpy by the ready removal of the wedge jaws and substitution of the interchangeable Charpy pendulum, but the additional uprights, scale and ball bearings necessary for a separate Charpy were very inexpensively put together. Although intended for a fixed height of release of 2 ft., an auxiliary adjustable release bar was provided for experimental work and a reverse extension on the pointer arm can be used in conjunction with a secondary scale to indicate the elevation of the striking edge in any starting position. This auxiliary release mechanism is shown in Fig. 3 in conjunction with the Charpy but it may be used with either machine. The primary scale is at present calibrated to show the actual height in feet to which the pendulum rises after breaking the specimen, as this scale is independent of height of drop and weight of pendulum and is thus adapted to use during the experimental period. Both these machines have been used rather extensively and found to be remarkably consistent, often indicating identical readings for a considerable number of samples. The Izod has been most used in our experimental work, first because Taylor, Davis and Wolf with whom results were being compared were provided with this type, and second because this machine as first constructed was provided with the variable capacity feature making possible the variation of energy of impact and velocity of impact independently of each other, thus allowing ready investigation of these factors both of which were thought to be of importance.

Comparison of Charpy and Izod Type Tests:

It was the thought of the committee interested that both the Izod and the Charpy type of test should be investigated as to their applicability to the insulating materials. The characteristics of both types of test have been brought out fairly well in the testing of metals, but with materials so different the usual objections to each do not necessarily apply. The Charpy is most favored in Continental Europe while the Izod is a British product and is also used quite widely in the United States.

The chief objection raised against the Charpy is the loss of energy resulting from friction of the specimen on the sides of the hammer, which may be considerable with ductile materials. This effect is not present in the Izod test, but for the same class of materials this machine has a more serious failing in that it has a maximum angle of bend of only about 60 deg. as compared with about 130 deg. for the Charpy so that in many cases materials sufficiently ductile as to be beyond the range of the Izod can be broken by the Charpy. For this reason, the Charpy is the more favored for adoption as a general test. However, neither of these features is of much interest as regards insulating materials which are as a class brittle and break with only a few degrees of bend.

Effect of Clamping.—On general principles, a test in which the specimen is clamped and so tested as to fail at the edge of the restraining surfaces is not good, due to the possible influence on the result by the clamping stresses. It was felt that this might be a pertinent objection to the Izod test in the case of the very weak and brittle molded materials, in spite of the dominating influence of the notch, and tests of three very different types of material were made to determine if any such effect could be distinguished. Duplicate lots of specimens were tested when clamped in the wedge jaws of the Bell Laboratories' Izod machine by the application of only 150 g. to a 3.5-in. rod in the capstan and when clamped as tightly as possible using the same rod. The impact figures obtained with this really tremendous difference in clamping pressure are given by Table I. The values shown are in each case averages of 3 specimens, the individual results being very consistent. Close agreement between individual specimens of the same lot is a marked characteristic of the test.

TABLE I.—EFFECT OF CLAMPING PRESSURE IN IZOD TEST.

	IMPACT STRENGTH, FT-LB.	
MATERIAL	SNUG CLAMPING	EXTREMELY TIGHT CLAMPING
Hard Rubber	0.260	0.264
Phenolic Cold Molded	0.149	0.155
Vulcanized Fiber, edgewise	1.09	1.04

The differences in the rubber and cold-molded materials are so slight as to be hardly significant and the increase in indicated strength with the loose clamping in the case of the fiber, while still relatively small, is most probably due to absorption of energy in motion of the specimen in the jaws. The snug clamping was obviously less than would be used in testing as it was not sufficient even to hold the specimen against the action of the ejector spring ordinarily used in these jaws and the tight clamping was such as to require a marked exertion. It is quite safe to assume that variations of clamping ordinarily encountered will be much less than these and, therefore, have no effect upon the Izod results for the materials with which we are dealing.

Data so far obtained indicate that the relative strengths of the different materials are roughly of the same order with either Izod or Charpy test leaving little to choose between them for testing the insulating materials. The results shown by Table II were obtained using the Charpy test with a span of 4 in. between supports of the specimen and the Izod which strikes the specimen 0.866 in. above the point of support.

The difference of conditions is most marked if we consider the total energy absorbed by the specimen as consisting of that part used in general deformation of the bar and that absorbed locally in the region of the notch. Because of the length of span the energy for general deformation is probably a larger part of the whole in the case of the Charpy which may explain the generally higher results shown for this test: The second factor probably explains the high value shown for the Charpy test of the phenol fiber tested flatwise. In the Izod test this material, tested in this direction, breaks with a ragged fracture extending $\frac{1}{8}$ to $\frac{1}{4}$ in. to either side of the notch with the shear planes at angles of about 45 deg., but in the longer span of the Charpy test the fracture area is multiplied many times, practically the entire specimen often being shattered by multiple fracture planes which undoubtedly are responsible for the unusual absorption of energy by this particular material in the Charpy test.

TABLE II.—COMPARISON OF CHARPY AND IZOD TEST.

(Averages of at least 4 specimens)

	IMPACT STRENGTH, FT-LB.	
	CHARPY	IZOD
Vulcanized or Hard Fiber, edgewise	1.21	1.13
Phenol Fiber, flatwise (laminated phenolic material, paper base)	1.267	0.64
Phenol Fiber, edgewise	0.546	0.30
Molded Fibrous Material	0.49	0.45
Hard Rubber, Grade A	0.286	0.25
Miscellaneous Molded Materials	0.18 – 0.21	0.13 – 0.17

At the present writing therefore it appears to be difficult to justify adoption of either Charpy or Izod test to the exclusion of the other except on rather arbitrary grounds such as convenience of specimen or the undesirability of continuing two tests. However, much can be learned by continuing both lines of investigation and this seems most desirable.

Effect of Weight of Machine:

In the impact testing of metals emphasis is laid upon the necessity of considerable weight of anvil to reduce to a minimum the absorption of energy in vibration of the testing machine, and, in fact, the difference between static resistance and impact strength has been sometimes attributed entirely to this vibration. As a consequence, every effort is made to make such machines heavy and substantial in construction. It is therefore interesting to compare the results of tests made with the quite light machines used by the writer and the heavier one used by Taylor. The Bell Telephone Laboratories' machines are of rigid construction and are clamped to a bench during tests but are of rather light weight, the total weight of the Izod being 45 lb. and that of the Charpy but 30 lb. The modified Ohio Brass Co. machine used by Taylor has a cast-iron base which alone is many times heavier than the whole weight of the small portable machine, but for tests made under comparative conditions the results are practically the same for both machines as shown by Table III.

TABLE III.—COMPARISON OF RESULTS ON LIGHT AND HEAVY MACHINES.

		IMPACT STRENGTH, FT-LB.	
NUMBER OF SPECIMENS		BAKELITE CORPORATION OHIO BRASS CO. HEAVY IZOD	BELL TELEPHONE LABORATORIES LIGHT IZOD
50	Molded Bakelite tested at velocities of 3.5 to 11.3 ft. per sec.	0.11 – 0.18	0.11 – 0.18
4	Phenol Fabric, flatwise, velocity 7.5 ft. per sec.	3.80	....
5	Phenol Fabric, flatwise, velocity 11.3 ft. per sec.	....	3.66
4	Phenol Fabric, edgewise, velocity 7.5 ft. per sec.	2.32	....
5	Phenol Fabric, edgewise, velocity 11.3 ft. per sec.	....	2.22
5	Phenol Fiber, velocity 7.5 ft. per sec.	0.576	....
10	Phenol Fiber, velocity 11.3 ft. per sec.	....	0.59

These results are in very close agreement, considering the rather radical differences in the two machines, and show no evidence of error due to absorption of energy in the small machine, as this would be evidenced by erroneously high readings, particularly in the case of the strong materials, whereas the actual differences are in the reverse direction. The differences of velocity shown are not of great effect in these materials and are of such a direction as not to affect the comparison. Tests made with both light and heavy pendulums in the light machine also show practically identical results. The close agreement of the light and heavy machines on the values of the low strength materials is the more remarkable when it is remembered that the pendulum weight of one machine is but one pound as compared with over seven pounds for the larger machine. Such close reading of the higher capacity machine is due in a large measure to the modifications of the pointer mechanism carried out by Taylor, as the Otis Elevator Co. was not able to obtain such accurate agreement with the machine in the original form. It may be concluded that the rigidity of the small machines is quite sufficient for the range of materials to be tested and that their accuracy in no way suffers because of their lack of greater weight. It is logical to expect that rigidity beyond a certain required minimum will have no measurable effect upon the result.

COMPARISON OF THE IMPACT TEST WITH STATIC FLEXURE TESTS

The essential property of resistance to failure under shock loading, which it is sought to measure by means of these machines, is essentially resistance to sudden fracture under rapidly applied, relatively high, localized stresses. The ability of a ductile material to withstand shock loading is best explained by its ability to safely equalize dangerously concentrated stresses by some plastic flow and from this it may be seen that the notch, as the cause of a concentration of stresses, is an essential feature of the test. The exact form of the notch is probably relatively unimportant provided the radius of the bottom is as small as practicable and the depth sufficient. Therefore for the purpose of the present investigation the form of the V notch most used in the work on metals was arbitrarily adopted. This is a notch having an included angle of 45 deg., a depth of 0.079 in. with the bottom rounded to a radius of 0.010 in. The samples used were of 0.5 by 0.5 in. cross-section, 2.5 in. long for the Izod test and 5 in. long for the Charpy and flexure tests. The notches were milled in all specimens equi-distant from both ends and in the case of some of the materials which varied from the 0.5 in. dimension the depth of the notch was varied to produce the same area back of the notch. The molded materials were so notched and tested that the blow was struck from a direction perpendicular to the action line of the pressure used in molding.

The question which naturally arises first in any investigation of the test is whether the presence of the notch alone, with the accompanying concentration of stress, will not give the desired information in the ordinary "static" flexure test. The total amount of energy used in breaking the specimen may, of course, be obtained from the stress-deflection curve and the relation, if any, between this value and that found in the impact test is of great interest. Therefore, duplicate lots of notched specimens were compared by the Charpy and the static flexure test. When the Charpy machine was built this comparison was kept in mind and the length of span, shape of specimen supports and striking edge were made the same as the corresponding features of the standard flexure test made in accordance with the Standard Methods of Testing Molded Insulating Materials (D 48 - 24) revised as recommended in 1925.[1] The comparison of results on both plain and notched bars in the Charpy test with a striking velocity of 11.3 ft. per sec. and the flexure test at a machine speed of 0.031 in. per min. is given by Table IV.

TABLE IV.—COMPARISON OF CHARPY RESULTS WITH TOTAL WORK OF FLEXURE TEST.

(Results are averages of at least 4 specimens)

Material Identification	Kind of Material	Charpy Test, ft-lb.		Flexure Test			
				Total ft-lb.		Maximum Load, lb.	
		Plain Bar	Notched Bar	Plain Bar	Notched Bar	Plain Bar	Notched Bar
1	Vulcanized Fiber, edgewise	....	1.22	5.0	1.95	330	239
2	Phenol Fiber, flatwise.....	2.87	1.27	2.2	0.25	441	164
3	Phenol Fiber, edgewise....	....	0.55	2.7	0.35	470	202
4	Molded Fibrous Material..	0.60	0.49	...	0.11	...	84.5
5*A*	Hard Rubber, Grade A....	1.70	0.29	10.0	0.53	247	124
6	Phenolic Hot Molded Material...............	0.652	0.20	0.46	0.09	214	87
7*A*	Phenolic Cold Molded A...	0.27	0.21	0.05	0.024	91	52.5
7*B*	Phenolic Cold Molded B...	0.23	0.20	0.04	0.02	75	36
8	Bituminous Cold Molded..	0.21	0.19	0.03	0.012	45	21.5
9	Shellac Mica Composition.	0.237	0.18	0.045	0.012	79	40

Table IV shows that the effect of the notch varies widely with different materials, indicating that the resistance of a mass of a given material to locally concentrated stresses may not be predicted from its behavior under the more or less uniformly distributed stress of the usual static test. These effects may not be attributed to the loss of "skin effect" as a result of breaking the surface of the material by milling the notch, as preliminary tests by Taylor indicate practically no difference in results obtained with milled notches and molded notches. Even resistance to concentrated stresses when slowly applied is not a measure of impact strength, as will be seen from comparison of the notched-bar results for Charpy and static tests. In fact, rapidity of application of load is undoubtedly a factor in the intensity of concentration of the stress. That the behavior of these materials is so widely different under these varying conditions seems definitely to indicate the existence of the so-called impact properties in the insulating materials.

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[1] 1924 Book of A.S.T.M. Standards and *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part I, p. 348 (1925).

EFFECT OF INITIAL ENERGY AND INITIAL VELOCITY OF IMPACT

Our earliest experience with the test showed that very different results were obtained with different initial elevations of the pendulum the strength apparently increasing with the initial elevation. This was thought to be due to the change of velocity of impact but change in the elevation of the pendulum increases the energy of impact as well as the velocity and it was necessary to separate the two factors to determine the true effect. This was done in a series of tests with

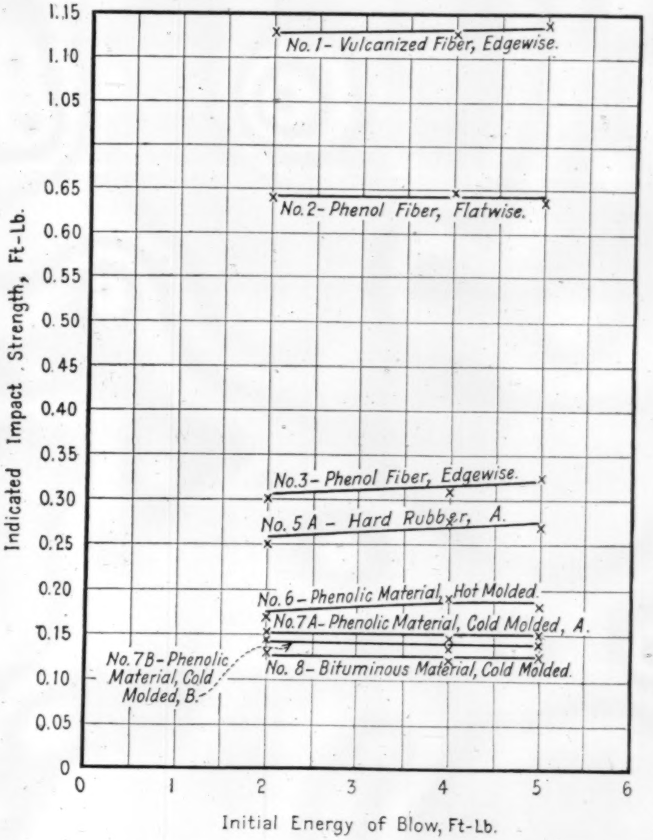

FIG. 4.—Relation of Initial Energy of Blow to Indicated Impact Strength with Velocity of Impact Constant at 11.3 ft. per sec.

the Izod machine which was designed to permit the variation of capacity by the addition of calibrated weights. Duplicate lots of certain of the materials listed in Table IV were tested with the velocity of impact the same (11.3 ft. per sec.) in all cases but using pendulums of weights corresponding to 2, 4 and 5 ft-lb. initial energy of blow.

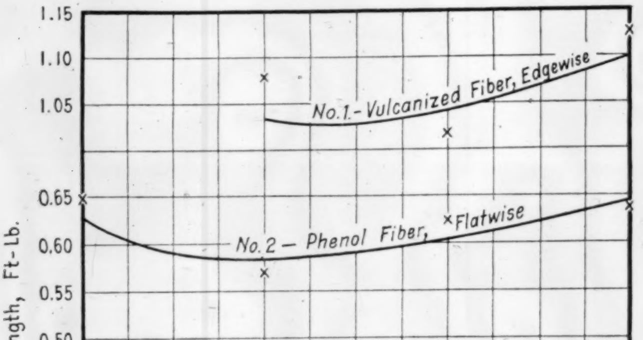

FIG. 5.—Relation of Velocity of Impact and Indicated Impact Strength.

From the results of these tests, shown by Fig. 4, it may be concluded that variation of initial energy over this range has no really significant effect upon the indicated values of impact strength though some slight increase with increase in energy might be expected from the resulting small increase in average velocity of fracture.

The velocity effect was further investigated by tests at velocities corresponding to initial elevations of the striking edge of 0.5, 1.0, 1.5

and 2.0 ft. with pendulum weights of both 1 and 2 lb. The relations of Fig. 4 were found to hold good at all velocities, the values for the 1 and 2-lb. tups with but one or two exceptions being practically identical over the entire range. The results, being so closely alike, are shown as averages for both pendulum weights in Fig. 5. This shows the effect of velocity of impact to vary with the material but being generally more important in the case of the weak molded materials and rubber than with the laminated materials. Whereas in the case of embrittled steels, increased velocity has been shown to cause a decrease in strength, the reverse is generally true of the insulating materials. The effect in the case of the molded materials is unmistakable, as an increase in energy absorption of 75 to 100 per cent accompanies an increase in velocity of impact from 5.6 to 11.3 ft. per sec. The error introduced by the practice of reducing the height of release in order to increase the sensitivity of the machine for tests of weak materials is from this apparent as the weak materials are made to appear relatively weaker and the strong relatively stronger and the conclusion that all tests must be made at a fixed velocity of impact cannot be escaped. Though the comparative results at different velocities of impact are shown by Fig. 4 to be substantially the same, the higher velocity is more desirable from a testing standpoint as it increases the strength figures of these very weak materials to a more easily measurable value and at the same time provides a more open scaling on the machine.

A number of interesting effects in connection with the tests, chiefly those involving the size of specimen and shape and dimensions of the notch, have not been explored as the present investigation has aimed only to determine the effect of the more important factors affecting the testing machine required.

CONCLUSIONS

The conclusions reached as a result of this work may be summarized briefly as follows:

1. For the insulating materials the comparative test gives practically all the information needed.
2. At the present writing, the Izod or Charpy type tests appear equally suited to the purpose.
3. Great weight of machine is not important for the capacity required provided the construction is substantial and rigid.
4. The insulating materials in general show impact properties not indicated by static tests, even of notched bars.

5. An increase of 150 per cent in initial energy of impact has practically no significant effect on the impact strength value.

6. For a true comparison of impact strengths a fixed velocity of impact is essential. The higher velocities are most desirable for mechanical reasons.

7. The notch appears to be an essential feature of the test, but further investigation of this point and the effect of various sizes and forms of notch is suggested.

DISCUSSION

Mr. Fogg. Mr. R. J. Fogg.[1]—I was interested to learn from Mr. Werring's paper that the manner of clamping the specimens in the machine had no effect on the values obtained. At the Fritz Engineering Laboratory, about two years ago, we made some impact tests on steel at low temperatures, running down to 40 and 50° F. below zero, and we got some very erratic results. We laid the irregularity of results to the method of clamping and I was interested to hear Mr. Werring say that he got the same results with loose clamping as he did when the specimens were tightly clamped.

I should like to ask whether when loosely clamped in the machine, the specimen rose as it was struck or whether it remained stationary?

Mr. Werring. Mr. W. W. Werring.—We did not notice any such rising effect though we attributed the slight variation in the case of vulcanized fiber to motion in the jaws under the light clamping, which was hardly any clamping at all. This was with extremely light clamping, when a force of only 150 g. at a 3½-in. radius was applied to the capstan. Due to the friction of the wedges this force just about closed the jaws on the specimen.

I hesitate to draw the definite conclusion that clamping will have no effect in the case of steel, as the forces usually involved are so much greater and conditions may be different, but certainly with our materials we could detect no such effect.

Mr. Fogg. Mr. Fogg.—At the low temperatures previously mentioned, our material became practically as brittle as glass. However, even under this condition, we got erratic results and at that time we charged it to the method of clamping.

Mr. Werring. Mr. Werring.—It might be possible that the irregularities in your test specimens were greater than those in ours, though I do not think we had ideal conditions as we had both milled and molded specimens in our tests. I should expect that the materials we listed as "cold-molded material" would show any clamping effect that existed, as these materials were really very weak and brittle and in our experience with apparatus parts are quite sensitive to irregularities in surfaces against which they are mounted.

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[1] Professor of Civil Engineering, Lehigh University, Bethlehem, Pa.

Mr. Buckingham.

MR. FRANCIS BUCKINGHAM.[1]—I am interested in Mr. Werring's statement about the light weight of the machine. We must keep in mind the very small capacity of the machine with which he worked; the maximum, I believe, was 5 ft-lb., so that the total weight of the machine (45 lb.) is practically ten times the energy of the blow. I do not think you will find any commercial impact machine, where the total weight of the machine is greater than ten times the energy of the blow. In the manufacture of impact machines we have found that if we have that much weight in the base it is a thoroughly stable machine.

Mr. Werring.

MR. WERRING.—I agree that the question of weight is only relative and that though my machines are of light weight the blow struck is light in proportion. The point I wished to bring out was not that lightness is desirable, but that rigidity beyond a certain minimum is not necessary and that the weight required in the case of the insulating materials is surprisingly small. The data on the subject were presented to emphasize the smallness of capacity required for the insulating materials and dispose of the conventional objection. The minimum weight machine for testing metals will of course be of great weight but for my purposes the usual heavy castings are unnecessary.

Mr. Smith.

MR. E. B. SMITH[2] (*by letter*).—In this paper Mr. Werring has presented further proof that the velocity of application of the impact force may have great effect on the results. In discussions in the meetings of the Impact Sub-Committee of Committee E-1, I have always maintained that this was one important factor which most experiments entirely ignore. Those tests that are sometimes quoted to prove the contrary have nearly always been on ductile materials that are more able to adjust themselves to distribute the stress. If a static test is greatly influenced by the speed of the testing machine,—less with ductile materials and more so with hard materials,—why is it not to be expected that some such effect, to a greater or less degree, may be observed for an impact test? Mr. Werring's tests further indicate the desirability of making impact tests under a standard rate of load application. The optimum rate will probably be different for different classes of materials. However, it should be chosen rather high, or at least such that the strength indications will have wide enough limits to afford good comparisons.

If impact test results were measured in terms of force instead of energy, we would measure more directly the resistance of a material to shock. Two different materials may absorb the same *energy* in breaking by impact, but the more ductile material because of its greater deflection will offer more of a cushioning condition to the blow, and the actual *force* (in pounds) developed will be less than for the harder material. The harder material will deflect less, the deceleration at impact may be greater, and the result may be a *greater force* developed. Since resistance to shock in most cases depends upon cushioning qualities, and upon cohesive strength, the material which withstands the greater *force* in impact is usually the more desirable for service under shock conditions. **Mr. Smith.**

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[1] Superintendent and Chief Engineer, Riehlé Brothers Testing Machine Co., Philadelphia, Pa.

[2] Research Professor of Mechanical Engineering, Iowa State College, Ames, Ia.

Mr. Whittemore. MR. H. L. WHITTEMORE[1] (*by letter*).—This paper presents the results of a conscientious effort to design an impact testing machine for insulating materials. In my opinion Mr. Werring is too much concerned because the results of an impact test, say on a new insulating material, cannot be used directly in design as the results of tension tests are used in designing structures. As he shows so forcefully, impact tests on materials show quality. The remarkably close agreement which he found between service tests and the results of impact tests is very gratifying.

Anyone who has developed testing apparatus can appreciate the labor which the design, building and testing of these impact machines involved. The machine shown in Fig. 1 of the paper seems unnecessarily expensive as there is considerable pattern work in this design. The frame shown in Fig. 2 is preferable on that account. It is highly desirable to keep the cost of testing equipment low and whenever practicable to design it so that it can be built in any machine shop. If this is done the number of manufacturers and users who test their materials as routine operation will be much greater than if the opposite course is followed. It seems evident that the entire community will benefit from an increase in the testing of materials.

The design of the pendulum arm for all these machines has apparently received considerable study, but no data are given on the stiffness nor upon how closely the actual velocity approached the computed velocity. Perhaps a stream-line tube connecting the tup with the axle would be cheaper and give as good results.

The results of the tests in the Bell Laboratories shows that comparing the results of Charpy and Izod tests there is little if any advantage in one over the other. It seems advisable therefore to standardize the tests, at least for testing material purchased under specifications, to prevent the development of a variety of specimens, machines, and

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[1] Chief, Engineering Mechanics Section, U. S. Bureau of Standards, Washington, D. C.

Mr. Whittemore. testing technique which it will be almost impossible to simplify later. This would of course be no bar to investigational work along any lines.

This paper is a valuable addition to our knowledge of the impact test and materials engineers will watch with interest the work being done by Mr. Werring and his associates, for their work on insulating materials may throw a new light on impact testing of metals.

Mr. Werring. MR. W. W. WERRING (*author's closure by letter*).—I agree with Mr. Whittemore on the desirability of simplified design of testing equipment and the Bell Telephone Laboratories machines were designed so as to involve only simple machine operations.

The pendulum arm was made as it was to insure maximum rigidity, though, of course, somewhat less rigid construction might suffice. The sawing out of the two halves from sheet aluminum was not difficult and the result is remarkably strong and rigid though I have no actual data on the subject.

As regards the losses in energy and velocity with these machines the conditions are as follows. The total bearing friction and windage on the down swing to the point of striking the specimen and picking up the pointer is 0.02 ft-lb. for the 2-ft-lb. tup and 0.03 ft-lb. for the 4-ft-lb. tup. The total loss of energy on the full free swing including pointer is very consistently found to be of the order of 0.05 to 0.06 ft-lb. A mathematically correct compensation based on observed values is applied to all results.

CAN THE HARDNESS OF MATERIALS BE NEGLECTED IN GRANULOMETRIC SIEVE ANALYSES?

By H. P. Hollnagel[1] and E. A. Harty[1]

Synopsis

In making granulometric analyses of soft materials, like talc, the authors find that the mechanical action of sifting breaks up the materials and that the test results are erratic. The length of time that the material is shaken, the kind of equipment and the methods employed should be standardized to permit duplication of results.

A granulometric analysis of a powdered material is usually made by shaking a certain amount of material through a stack of standard mesh sieves. This shaking is done either by hand or mechanically, if the necessary equipment is available. As a consequence of the process of shaking, the powdered material is caused to tumble back and forth on the several sieves and gradually works through the wire mesh on to the next sieve below. This operation is continued for a time, depending upon the quantity of material that is to pass through the stack of sieves, and the individual making the analysis.

What happens when a material like talc is subjected to a rubbing effect like that which occurs when the material is tumbled back and forth on the bronze wire mesh of a stack of sieves?

Recently, the authors had occasion to make a granulometric analysis of various samples of this material. Standard 8-in. Tyler sieves and a Ro-tap machine were used. Twenty grams of talc were placed on the coarsest sieve (No. 50) and shaken for 5 minutes. Each sieve with its contents was then carefully weighed and after subtracting its weight when empty the net amount of the residue was obtained. The results, when expressed in cumulative per cent are an indication of the analysis of that specimen after 5 minutes. The sieves with their respective residues were again placed on the machine and shaken for an additional 10 minutes and the same procedure followed. In this manner, the analysis of two samples, A and B, for 5, 15, 30 and 45 minutes was obtained.

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[1] Thomson Research Laboratory, General Electric Co., West Lynn, Mass.

The results are shown in the curves represented in Fig. 1, in which the ordinates represent cumulative percentage of material retained, and the abscissas the sieve numbers. In view of the fact that these tests were made with sieves having meshes 50, 100, 150, 200, 250 and 300, respectively, the lines joining the respective percentages should not be considered too seriously as representative of amount

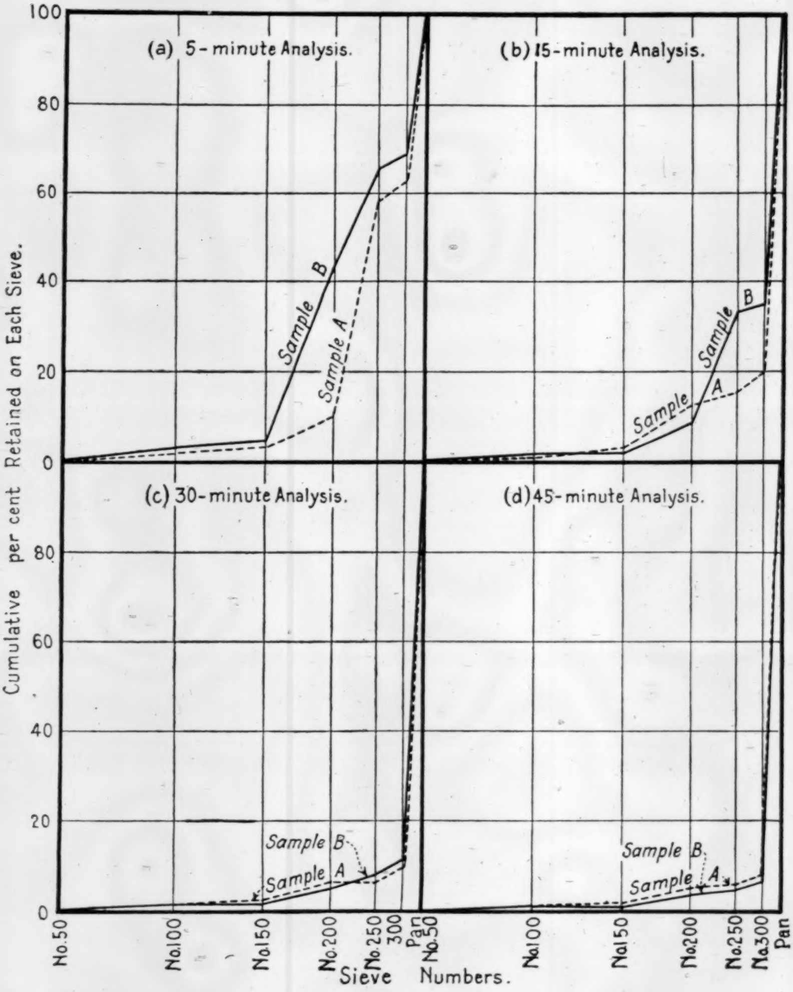

FIG. 1.—Granulometric Analysis of Two Samples of Talc Sieved for 5, 15, 30 and 45 Minutes.

accumulated on the meshes which lie between the several numbers represented in these tests. It can be said, however, with reasonable assurance that these graphs represent average conditions.

Fig. 1(*a*) shows a 5-minute analysis of two different samples designated as A and B, the latter apparently containing a higher per-cent residue on each sieve. Fig. 1(*b*) shows the result of a 15-minute analysis of these two samples. Their characteristic curves now cross each other and the per cent residue is higher for sample A up to 200 mesh, although in Fig. 1(*a*) sample B appeared to have the highest. Fig. 1 (*c*) shows a 30-minute analysis of the two samples wherein sample B apparently has a greater residue on 250 and 300 mesh than does sample A. Fig. 1(*d*) indicates a 45-minute analysis of them, sample A appearing to be coarser, although at the outset it was the finer of the two talcs.

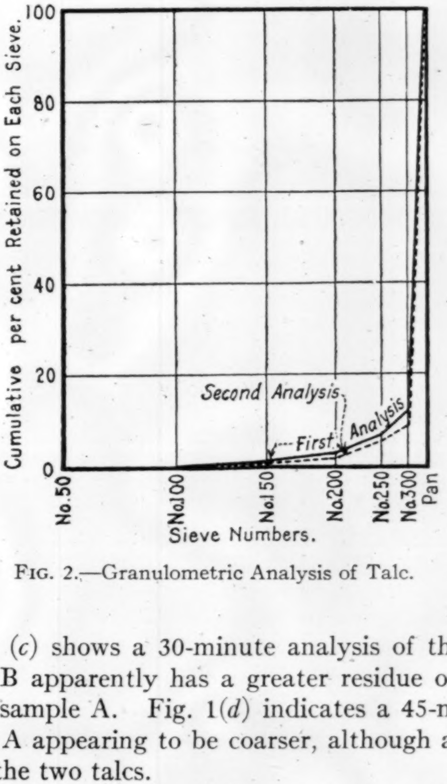

FIG. 2.—Granulometric Analysis of Talc.

These tests were repeated several times and lead to the conclusion that the continuous rubbing of talc on the bronze wire causes it to crumble, becoming finer and finer, so that if the operation of shaking were continued over an extended period, all material would ultimately go through the finest mesh sieve, that is, No. 300. To substantiate this conclusion, a quantity of the same talc was shaken for a long time and upon examination, it was found that a constant residue could not be obtained.

Other tests were then made as follows: 20 g. of talc were shaken for 15 minutes and its cumulative analysis in per cent recorded. The residues were carefully saved and mixed up and turned over to a third party[1] for analysis. These were shaken for 15 minutes and the cumulative per cent recorded. Fig. 2 shows the comparison of these two analyses, which proves that the talc actually breaks up, as the same sample gives two distinct analyses.

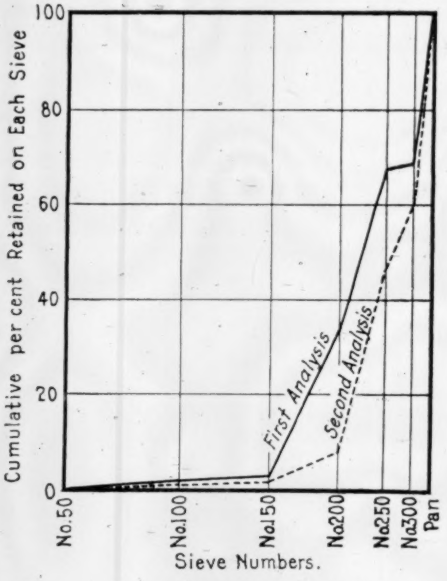

FIG. 3.—Granulometric Analysis of Talc.

Another sample of talc was shaken for 5 minutes in the same stack of sieves and its cumulative analysis in per cent recorded. The same sample was then shaken again in these sieves for 30 minutes and the cumulative analysis in per cent recorded. The residues on the several sieves were thereafter mixed together and again placed on the coarsest sieves of the stack and another 5-minute analysis made. These results are plotted in Fig. 3 and show that the second 5-minute analysis is quite different from the first. This is probably due to the breaking of the talc, particularly since the entire quantity had practically passed through all the sieves in 15 minutes, the time previously required being 30 minutes.

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[1] Through the courtesy of Mr. J. M. Darke and Mr. L. B. Mears of the Materials Testing Laboratory, General Electric Co., Lynn, Mass.

From the results of these tests, it appears that a soft material like talc, which is recognizedly at the bottom of the mineralogical hardness scale, should not be sifted on a bronze sieve, whose hardness is greater, without some understanding with regard to definite standards. While the logical procedure would be to sift substances in sieves made of the same material as those whose granulometric analysis is required, this is quite impossible in the majority of cases for self-evident reasons. It would also require a multiplicity of equipment quite unnecessary. A better method of procedure would be a standardization for this kind of analysis. In the opinion of the authors, the establishment of a standard type of equipment, together with a proper determination of the times of shaking, would be a step in the proper direction.

While the tests herein described hold for the materials of lower hardness, exactly the reverse conditions would exist were like sieves used on materials having greater hardness though the time element involved would be quite different as recorded in the wear of the bronze sieve.

In conclusion, it may be remarked:

1. That materials of different degrees of hardness should be shaken for different lengths of time.
2. As a consequence of the difference in hardness between screen and material, either the material or the screen must necessarily wear. Charts are shown depicting what happens in the case of the analyses of talc.
3. Some sort of standardization should be sought, with reference to the manner and time in which such analyses should be made.
4. That such standardized screens should be examined microscopically from time to time to determine whether the proper mesh ratios are being maintained.

A NEW TYPE OF MIRROR EXTENSOMETER

By M. F. Sayre[1]

Synopsis

This paper describes a mirror type extensometer adaptable to routine testing of tension test specimens of 2 or 8-in. gage length, where a high degree of precision in proportional limit or modulus of elasticity determinations is desired. By use of a system of four mirrors, two ends are gained: (1) elongations on both sides of the specimen are automatically averaged, and (2) any movement or vibration of the specimen is automatically cared for, so that readings are unaffected.

By use of a "crossed spring" support, it becomes possible to attach the movable mirrors permanently to the main body of the extensometer, eliminating need for the separate Martens lozenges, and so making the extensometer as a whole much simpler to handle.

Readings may be taken directly to 1 part in 100,000 on a 2-in. gage length, and by estimation to a fraction of that figure.

Mirror extensometers have been regularly used in the past when it was necessary to obtain elongation measurements to the highest possible degree of precision, but it has been difficult to combine this high degree of precision with convenience of operation, and so their use has generally been restricted to research, rather than production testing.[2]

During the past few years, the author has had occasion to make a large number of tension tests on various non-ferrous alloys. Most of these had very low proportional limits, so that the unit strain at the proportional limit was generally below one part in a thousand and sometimes below one-third of that figure. With such low elongations, high precision in extensometer measurements was called for, and it became worth while to investigate whether it would not be possible to develop an optical type of instrument for use on the standard specimens of 2-in. gage length which could be manipulated rapidly enough to put its use at least on a "semi-production" basis.

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[1] Associate Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[2] An exception is the "Wire Testing Extensometer" described by Messrs. T. D. Lynch and P. H. Brace before the American Society for Testing Materials in 1919 (*Proceedings*, Am. Soc. Testing Mats., Vol. XIX, Part II, p. 696 (1919)).

The resulting instrument is shown in side view in Fig. 1. Optically, it consists of a telescope with horizontal cross-hair, supported either on a tripod or on a bracket attached to the testing machine, a system of four mirrors on a light-weight extensometer attached to the test specimen, and a graduated scale supported at a fixed distance from the test specimen on the opposite side of the specimen from the telescope. The line of sight turns through an angle of 90 deg. at each mirror, emerging practically in line with its original direction. The

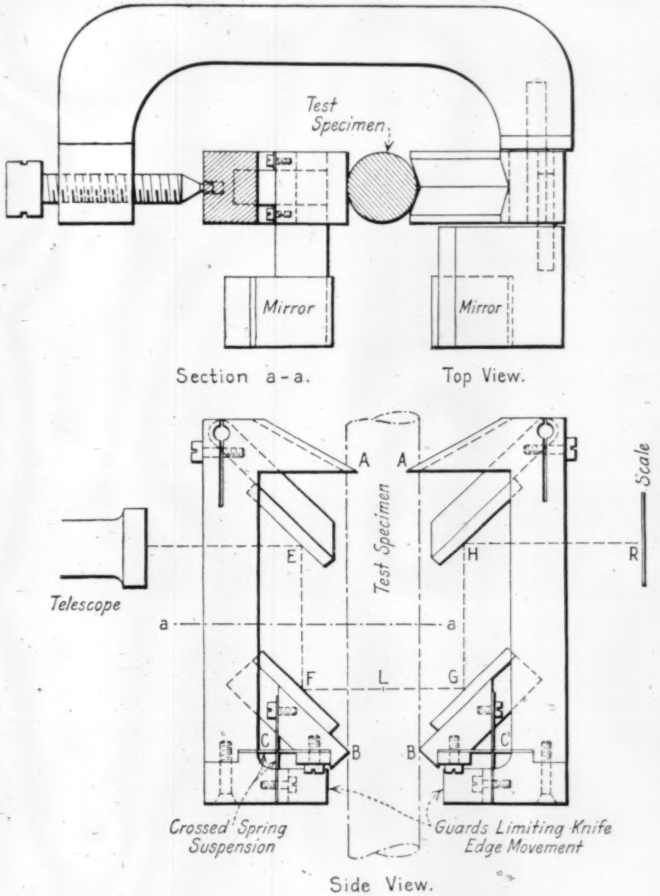
Test Specimen
Mirror
Mirror
Section a-a.
Top View.
A
A
Scale
Test Specimen
E
H
R
Telescope
a
a
F
L
G
C
B
B
C'
Crossed Spring Suspension
Guards Limiting Knife Edge Movement
Side View.

FIG. 1.—Mirror Extensometer

Fixed knife edges at *A*. Movable knife edges at *B*. The lower mirrors pivot about points *C* and C′ at intersection of crossed springs.

Line of sight passes one inch to left of center of test bar.

upper two mirrors are fixed in position during any one test, but may be tilted slightly back or forth to allow for varying positions of the telescope and scale. The lower mirrors are so supported that they tilt as the test bar elongates.

This system of mirrors offers great practical advantages in that, first, it automatically averages the elongations on the two sides of the bar, thus avoiding the use of two telescopes and two scales, with two sets of readings to be averaged, as with the original Martens type; and second, it makes the reading almost immune from the effect of any movement or vibration of the test specimen or testing machine. Any up and down, or sidewise movement of the test bar as a whole, within reason, does not affect the scale reading at all. An angular tilt of the specimen changes the reading, but only by an amount equal to the sine of the angle turned through, multiplied by the sum of the two short distances, EF and GH. For any likely angular twist, this amount is negligible.

As load is applied to the test specimen, the distance AB increases, and the mirrors pivot about C and C', turning through an angle α such that $\alpha = \frac{\text{elongation}}{\text{distance } BC}$. The line FG turns through an angle 2α and GH and HR each through angles of 4α. As a result, the reading changes by an amount

$$GHR \times 4\alpha + FG \times 2\alpha$$

or more conveniently,

$$LGHR \times 4\alpha,$$

the point L being opposite the center of the test bar.

Making $BC = 0.5$ in. and $LGHR = 250$ in., we have that

$$\text{Elongation} = \frac{\text{scale reading in inches}}{2000}.$$

At the start, either the telescope or the scale is shifted so that the initial reading is zero, so eliminating any index correction. With scale divisions of 0.04 in., each division corresponds to an elongation of a fifty-thousandth of an inch (0.00002 in.), or 1 part in 100,000 on a 2-in. gage length. The distance HR could of course be made larger or smaller as desired, within the focusing limits of the telescope used, and the scale be graduated accordingly. It has proved to be easily possible to estimate fractions of a scale division, and so read down to possibly 1 part in 500,000 on a 2-in. gage length. To do this the scale has had to be properly illuminated, using one or two electric lights close to the scale.

A second feature which contributed to the success of the instrument has not yet been mentioned. The use of a Martens lozenge as a support for the movable mirrors makes a very simple construction possible, and the use of a small size of lozenge permits of a high multiplying factor for the optical lever. On the other hand, it is difficult or impossible to fasten the lozenges and attached mirrors to the frame of the extensometer, so that there are two extra parts to be handled, making it virtually a two-man job to put the extensometer together in place. In place of the lozenges, the two movable mirrors were supported by flat steel plates, one corner of which formed the knife edge at the specimen. These plates were attached to the frame by means of horizontal and vertical springs. These flat springs were flexible enough to allow angular movement of the plates, but controlled the movement so as to compel the plates to rotate about virtual centers at C and C', the lines of intersection of the two springs. The crossed springs are of course staggered, with the vertical spring in the middle and two narrower horizontal springs, one on each side, so as to avoid interference. For all small angles of rotation, this virtual center remains essentially fixed in position, making this device the equivalent of a Martens lozenge with effective length BC. These springs automatically hold the two knife-edges A and B at the proper distance apart, so as to give the correct initial gage length. For the suggestion of this general method of support, Prof. Earle Buckingham,[1] now at Massachusetts Institute of Technology, is responsible.

The light screw clamp used to hold the extensometer to the specimen is shown in the top view of Fig. 1. This clamp is loosely attached on one side and has a thumb screw which bears on the other side, with sufficient range of adjustment to care for specimens from a minimum thickness of a few hundredths of an inch to a maximum of 1½ in. diameter.

The mirrors and all delicate parts of the extensometer are protected from damage by an aluminum guard, not shown in the figure. A stop, which is shown in the figure, limits the movement of the lower mirror so as to prevent breakage of the springs.

The telescope used may be virtually any instrument which can be held firmly, and which is equipped with a horizontal cross-hair. For our work, the most easily accessible instrument proved to be an engineer's transit. This was supported on a bracket clamped to the frame of the testing machine, which was made adjustable in height to allow for varying lengths of test bar, but was permanently fixed in the proper position horizontally.

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[1] This same crossed-spring type of support was used by Austin Mesnager in an extensometer described by him in *Annales des Ponts et Chausses*, in 1903. This extensometer was of the compound lever type.

Only one instrument of this type has been made, but this has been in use for the past two or three years on some hundreds of tests. Difficulty was experienced at first in obtaining a clear sharp image of the scale through the system of four stellite mirrors. This proved to be due mainly to the fact that the mirrors were clamped down too rigidly, and hence became slightly warped. A second difficulty was caused by the fact that the mirrors themselves were made too small, only $\frac{7}{16}$ in. wide. As a result, much time was wasted in juggling the telescope back and forth so as to find the image of the scale in the little mirrors. The use of larger mirrors or possibly, better, of prisms $\frac{7}{8}$ or 1 in. square, would avoid most of this difficulty.

Aside from these two difficulties, the instrument has proved to be very satisfactory. The precision obtained was all that could be desired; the readings could be taken very quickly, if necessary without stopping the testing machine, and gave the unit strain directly, without computation. With the spring connections, backlash is very nearly eliminated. This factor had given a good deal of trouble when dealing with the different types of extensometer which use a mechanical lever system. A hundred-thousandth of an inch, or even a ten-thousandth, is not a large distance, and either a slight play in pivot points, if loose, or on the other hand a binding action at the same points, if tight, may introduce that much error. This becomes particularly important when any attempt is made to determine the true elastic limit by removing the load and measuring permanent set.

Certain fundamental theoretical errors exist. These are practically the same as for the Martens type extensometer, and have been sufficiently discussed elsewhere. For the range of elongation usually measured, their effect is extremely small, in fact negligible. A little care must be exercised to see that the lower knife edges are in normal position, that is, at exactly 2 in. from the upper gage points, at the start of the test. This is necessary both to obtain the correct initial gage length and to partially eliminate the theoretical errors already mentioned. A more important error is probably the effect of temperature changes. For precise work, the instrument should be shielded from direct draughts, and both instrument and test specimen should be at equilibrium temperature during the run.

DISCUSSION

Mr. McVetty.

MR. P. G. MCVETTY[1] (*presented in written form*).—During the past few years, the subject of methods of determining proportional limits has received considerable attention. It is indeed interesting, therefore, to find that the mirror type of extensometer has been put into convenient form in order to adapt it to the field of routine testing. Up to this time, the limit of commercial accuracy of strain measurement of standard 2-in. test specimens has been of the order of 0.0001 in. per inch. For precision testing, the Martens extensometer reduces this limit to 0.000002 in. per inch, and the order of accuracy of the instrument here described is similar. The adaptation of this instrument to routine testing involves the measurement of extensions one-fiftieth as great as the smallest now commonly determined. A change of this magnitude requires some explanation to show under what conditions this degree of refinement is necessary or desirable.

Tests reported by D. J. McAdam[2] and by N. L. Mochel[3] indicate that the observed values of proportional limit, elastic limit and Johnson's limit for some materials show considerable variation at normal temperatures. The difficulty of determining these values for any material increases as the temperature is increased. It follows that certain alloys at normal temperature and materials in general at elevated temperatures require precision methods of tension testing if accurate values are required. A study of actual test curves shows that the instrument here described should give an apparent value of proportional limit of normalized medium-carbon steel at 400° C. about 17 per cent lower than could be observed with an extensometer having a sensitivity of 0.0002 in. A similar comparison for heat-treated stainless iron shows a difference of more than 50 per cent. A proposal to change the order of accuracy of strain measurement in routine tests should be advanced with the understanding that proportional limit values so obtained are not directly comparable with those obtained with the usual commercial extensometers. This is especially true when a designer collects test data from various sources and attempts to decide the relative value of different materials for some application at elevated temperatures.

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[1] Mechanical Engineer, Research Department, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] D. J. McAdam, Jr., "Endurance Properties of Corrosion-Resistant Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 273 (1924).

[3] N. L. Mochel, "Stainless Iron," *Transactions*, Am. Soc. Steel Treating, Vol. X, p. 353 (1926).

Mr. McVetty. Another factor which requires consideration is the sensitivity of the testing machine itself. The relation between the required sensitivity of stress and strain measurement varies with the modulus of elasticity of the material, as follows:

	Modulus of Elasticity, lb. per sq. in.	Order of Accuracy of Strain Measurement, in. per inch	Corresponding Sensitivity of Testing Machine Required, lb. per sq. in.	Total Load on Standard 0.505 in. Diameter Test Piece, lb.
Steel..........	30 000 000	0.000100	3000	600
		0.000002	60	12
Aluminum.....	10 000 000	0.000100	1000	200
		0.000002	20	4

The sensitivity of most testing machines is such that differences of 200 lb. or less are easily measured, but in many cases it is doubtful if a difference of 12 lb. can be indicated. An examination of the table shows that we are justified in using extensometers which read closer than 0.0002 in. for routine testing, but we may not be justified in going to the limit of 0.000004 in. This depends upon the size and the condition of the testing machine and it must be considered especially when tests involving small loads are made in a large machine.

The extensometer described by the author has the advantage of sensitivity equivalent to the Martens apparatus with the additional advantage of freedom from effects of vibration. The latter would allow its use in many places where Martens readings could not be obtained on account of vibration. It will be noted that the distance from the test specimen to the scale is about 20 ft. instead of 3 ft. as in the case of the regular Martens apparatus. In some cases, this distance would be inconvenient and any decrease results in a proportional decrease in the sensitivity of the instrument.

The main disadvantage of this instrument appears to lie in the fact that "elongations on both sides of the specimen are automatically averaged." This prevents comparison of readings on the two sides to detect evidence of eccentricity of loading. This often occurs and the actual stress is a combination of tension and bending stresses of which the former alone is measured. The present standard method of test allows a deviation from correct alignment of 0.01 in. The accompanying Fig. 1 shows that this amount of eccentricity of loading on a standard $\frac{1}{2}$-in. test specimen produces enough bending to make the maximum stress about 15 per cent greater than the average. In all cases, the measured stress is lower than the actual stress which produces deviation from proportionality. We have had cases in which

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Mr. McVetty.

this error amounted to 40 per cent and it is our practice to discard determinations of proportional limits in which the readings show evidence of eccentricity of loading.

In conclusion, the following points should be noted:

1. Precision extensometers must be used in tests for proportional limits at elevated temperatures and, in the case of some materials, at normal temperature also.

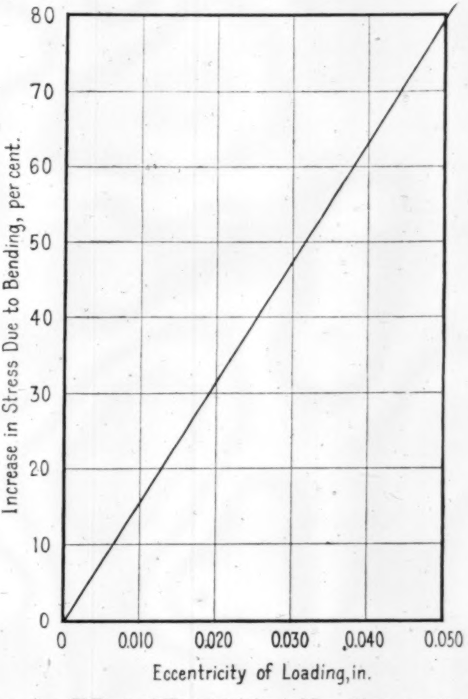

FIG. 1.—Effect of Eccentricity of Loading on the Determination of Proportional Limit in Tension, Using Standard Tension Test Specimen 0.505 in. in Diameter.

2. There is a definite relation between the requirements of accuracy of stress and strain measurement in the determination of proportional limits.

3. It is useless to expect precision results from refined strain measurements unless the testing machine is capable of similar accuracy.

4. Testing machines used for routine tests are not generally capable of the sensitivity which justifies extreme refinement of strain measurement.

Mr. McVetty. 5. Where precision is required and a suitable testing machine is available, there is a definite need for a simple, sensitive extensometer which is not affected by vibration and which is capable of detecting eccentricity of loading.

6. The instrument described by the author combines most of these requirements, but the one fact that eccentricity of loading may exist without possibility of detection prevents its use for precision determinations of proportional limits.

Mr. Sayre. MR. M. F. SAYRE (*author's closure by letter*).—Mr. McVetty's discussion brings up a number of points with which I would heartily agree, and a few with which I would dissent.

The sensitivity of the testing machine used should of course bear a reasonable relation to the precision of the strain gage readings. Any ordinary commercial testing machine in reasonably good condition, and with capacity not unduly great in comparison to the strength of the test specimen, will certainly be amply sensitive to justify readings to 0.00001 in. per inch. This corresponds to a sensitivity of 60 lb. with steel, or 20 lb. with aluminum samples, with $\frac{1}{2}$-in. diameter test specimens. Using the mirror type of extensometer and 2-in. gage length 0.00001 in. per inch should represent one scale division, and readings would be made very rapidly, without any need to estimate fractions of a division. Much of my own work has been done simply to that degree of precision. For other work, particularly where alloys with very low proportional limits were dealt with, a 5000-lb. capacity testing machine was used, sensitive to one or two pounds and probably accurate within five or ten pounds. Extensometer readings were made to one fifth of a scale division, requiring only slightly more time for reading.

Some misunderstanding may have arisen from the fact that the distance from the test specimen to the scale was mentioned as 250 in. This length was chosen simply because it fitted conditions in our laboratory. Within reasonable limits, the sensitivity is independent of the length used, provided the scale used is graduated to suit the distance. The finely divided scale at 30-in. distance occupies exactly the same space in the telescope field of view as the coarse scale at 250-in. distance, and is read in just the same way. It may be worth noting that instruments are now being made up, modified in design to eliminate some of the inconveniences mentioned in the original paper, and that these instruments have scale distances of 30 to 36 in.

Eccentricity of loading is an important factor in any precision testing, and test bars must be so shaped and so gripped as to bring

Mr. Sayre. it to a minimum. However, the following modifying factors should be noted:

1. In test specimens turned to size in a lathe, the eccentricity of shape should be of the order of a thousandth of an inch, rather than a hundredth of an inch. The resultant increase in extreme fiber stress would be 1.6 per cent, not 15 per cent.

2. The increase in stress due to the bending moments induced by improper gripping of the specimen may be far larger, up to 15 per cent or more. The direction in which this error should be attacked is obvious.

3. The effect of these computed increases in stress upon the test results is far less than these figures would indicate. Except in very brittle materials, the ultimate load on the test specimen is but little affected. The apparent proportional limit will usually be changed by only half or a third or a quarter as much as the change in nominal extreme fiber stress, the exact effect depending upon how sharply the true stress-strain curve breaks away from a straight line in the neighborhood of the proportional limit. Modulus of elasticity will be unchanged.

It is naturally desirable while making the extensometer readings, to be able to detect the presence of these undesirable bending stresses in the test specimen. It is, however, a question as to how far convenience and speed of operation should be sacrificed in order to gain this ability. Even with the usual Martens extensometer, this ability is only partially obtained, as it indicates bending moments about the plane of the mirrors only. Internal stresses due to bending moments about a plane at right angles to this may exist without detection. In the one plane, the results are not certain, as a tilt of the specimen as a whole will have the same effect upon the readings for the two sides of the specimen as will an eccentricity of loading, and may either mask or exaggerate the effect of the eccentricity. An extensometer could readily be designed which would operate to detect all eccentricity of loading, but it would be more complex than the Martens, and would involve reading more than two scales. For very precise work, and particularly as a means of investigating the effectiveness of various methods of gripping test bars, so that internal stresses may be avoided, this higher degree of refinement will be justified.

A NEW TWIST METER FOR TORSION TESTS

By J. Hammond Smith[1]

Synopsis

This paper describes a new type of instrument for accurately measuring the twist in torsion tests. The instrument is designed in such a manner as to be quickly attached to and removed from test specimens. It has an accurately fixed radius of arc, on which the twist is measured to one-ten-thousandth of a turn. The effect of bending in the specimen is eliminated from the twist readings.

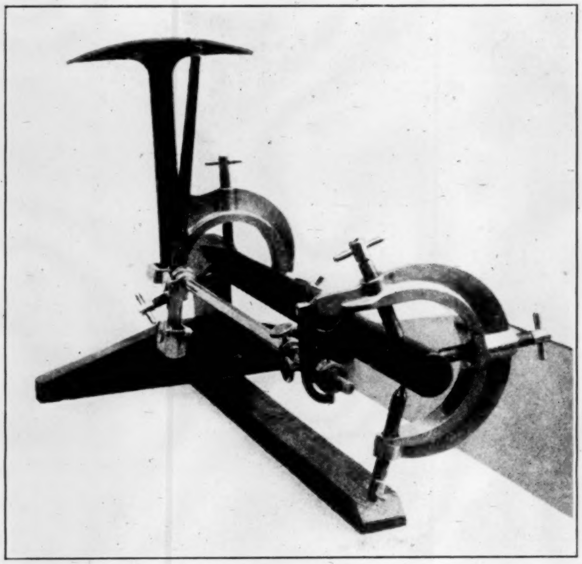

Fig. 1.—Twist Meter for Torsion Tests.

Description of the Instrument

The desire to obtain quickly and conveniently accurate readings of twist in torsion tests prompted the design of the instrument described in this paper. The main objects to be attained are: facility in attaching and detaching the instrument; an accurately graduated arc and vernier of fixed radius which requires no adjustment when

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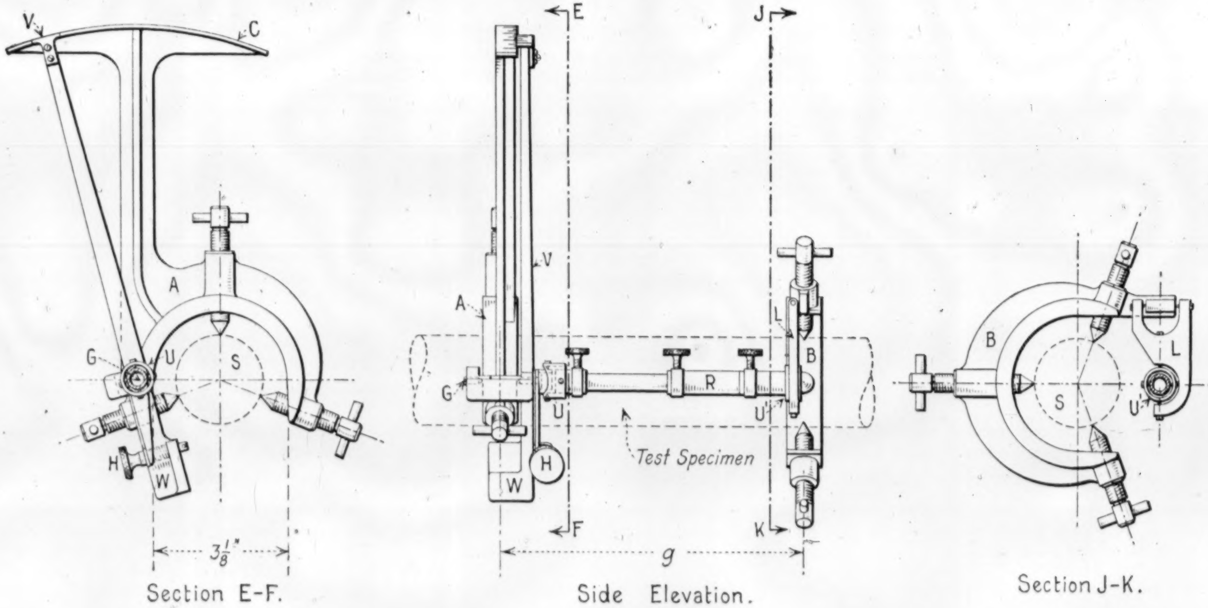
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Side Elevation.
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Section J-K.

FIG. 2.—Diagram of Twist Meter for Torsion Tests.

attaching the instrument to a specimen; and the elimination of the effect on twist readings of bending in the specimen.

As designed, the instrument may be used on specimens ranging in size from ½ to 3 in. in diameter, and of almost any length. It is illustrated in Fig. 1.

TABLE I.—RESULTS OF TORSION TESTS.

Seamless Steel Tubing (S. A. E. steel 1040). Outside diameter, 3 in. (No. 13 B. w. g.).

Load, in-lb.	Twist, turns in 36 in.				
	Specimen No. 1, Quenched at 1500° F. Drawn a 930° F.t	Specimen No. 2, Quenched at 1500° F. Drawn at 1030° F.	Specimen No. 3, Quenched at 1500° F. Drawn at 1130° F.	Specimen No. 4, Quenched at 1525° F. Drawn at 1200° F.	Specimen No. 5, Quenched at 1525° F. Drawn at 1250° F.
0	0	0	0	0	0
2 000	......	......	......	......	0.0005
4 000	......	......	......	......	0.0010
5 000	0.0015	0.0012	0.0011	0.0013	......
6 000	......	......	......	......	0.0016
8 000	......	......	......	......	0.0021
10 000	0.0028	0.0025	0.0026	0.0028	0.0028
12 000	......	......	......	......	0.0032
14 000	......	......	......	......	0.0038
15 000	0.0041	0.0038	0.0039	0.0042	......
16 000	......	......	......	......	0.0044
18 000	......	......	......	......	0.0050
20 000	0.0055	0.0050	0.0051	0.0057	0.0056
25 000	0.0068	0.0064	0.0066	0.0071	0.0071
30 000	0.0082	0.0076	0.0078	0.0086	0.0087
35 000	0.0098	0.0088	0.0091	0.0101	0.0103
40 000	0.0111	0.0100	0.0105	0.0117	0.0117
45 000	0.0126	0.0112	0.0119	0.0133	0.0133
50 000	0.0140	0.0124	0.0132	0.0150	0.0150
55 000	0.0153	0.0136	0.0147	0.0170	0.0171
0	0.0003	0.0002	0.0001	0.0014	0.0014
55 000	0.0153	0.0137	0.0145	0.0176	0.0175
57 000	......	......	......	0.0183	0.0181
60 000	0.0167	0.0149	0.0158	0.0197	0.0194
62 000	......	......	......	0.0209	0.0204
65 000	0.0181	0.0162	0.0170	0.0228	0.0220
67 000	......	......	......	0.0245	0.0233
70 000	0.0196	0.0177	0.0184	0.0275	0.0254
73 000	0.0205	0.0188	......	0.0318	0.0281
75 000	0.0212	0.0199	0.0198	0.0370	0.0304
77 000	0.0219	0.0210	0.0232	0.0430	0.0340
80 000	0.0230	0.0231	Collapsed	Collapsed	0.0387
82 000	0.0238	0.0249	......	......	Collapsed
85 000	0.0250	0.0286	......	......	......
0	0.0021	0.0074	......	......	......
85 000	0.0253	Collapsed	......	......	......
90 000	0.0272	......	......	......	......
100 000	0.0339	......	......	......	......
105 000	0.0392	......	......	......	......

The three views of Fig. 2 serve to make the construction of the instrument clear. The yokes *A* and *B* are attached to the specimen *S* at the desired spacing *g* by means of the hardened point contact screws shown in the figure. The adjustable shaft *R* is clamped in position between the yokes, with the vernier *V* set near zero of the graduated arc *C*. When ready to start the test, the zeros of arc and vernier are made to coincide by means of the zero adjusting screw *H*. Thus the readings will start at zero load and zero twist. Vernier *V* is pivoted on the frame of yoke *A* at *G*, the center of arc *C*, and thus while moving along the arc it rotates in a plane approximately perpendicular to the axis of the specimen. The vernier arm is counterbalanced by means of the small fixed weight *W*. The zero adjuster is centered on the axis *G* of the vernier arm, and carries the universal coupling *U* which connects directly to the adjustable telescoping shaft *R*. The body of yoke *B* is similar in construction to that of yoke *A*. It carries a swinging link *L* which carries universal coupling *U'*, connecting with shaft *R*.

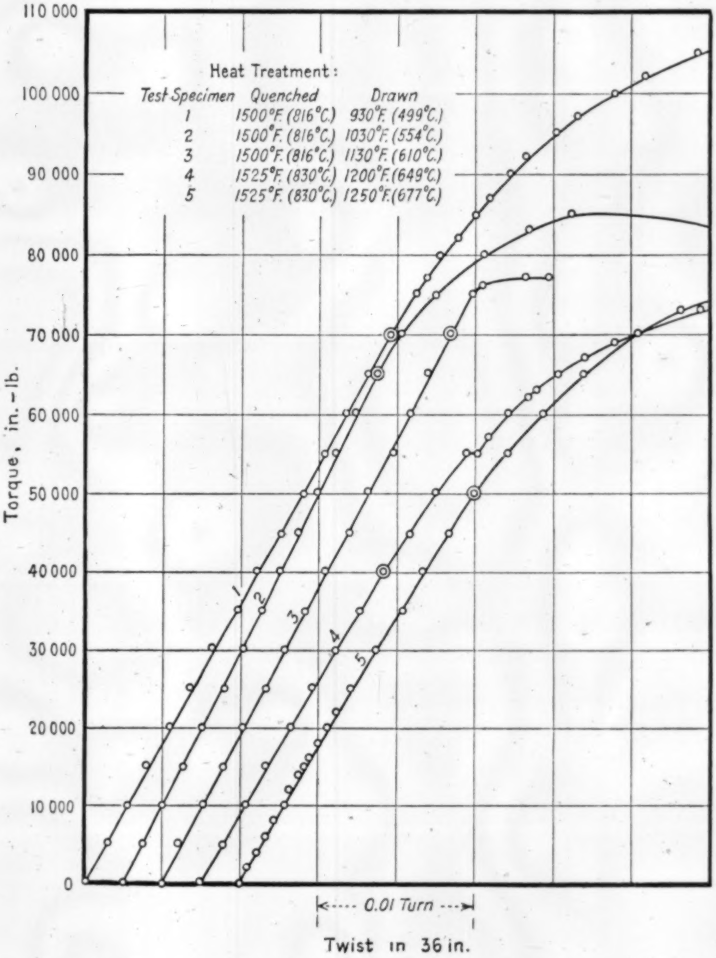

FIG. 3.—Showing Results of Torsion Tests on Seamless Steel Tubing (S.A.E. 1040). Outside Diameter, 3 in. (No. 13 B. w. g.). Gage Length of Twist Meter, 36 in.

Thus any relative rotation of the test specimen between yokes *A* and *B* is transmitted through this intermediary coupling between the yokes to the vernier moving on the graduated arc. The only motion transmitted between the yokes is that of rotation. Errors in twist readings ordinarily caused by bending in the test specimen are entirely eliminated by the universal couplings of the yoke-connecting shaft. The couplings are properly oriented in relation to each other to secure greatest accuracy in movement.

The arc is graduated in turns, the smallest division being one-one-thousandth of a turn. The smallest direct reading from the vernier is one-ten-thousandth of a turn. We find this to be the most convenient graduation to use. Readings may be quickly reduced to degrees or radians by the multiplying factors 360 or 2π, respectively.

RESULTS OF TESTS

This type of instrument has given excellent results on a large variety of torsion tests. It has also been used on special work, such as the determination of twist in beams when eccentrically loaded.

Table I and the corresponding curve sheet of plotted data (Fig. 3) are submitted as samples of results obtained with this instrument. They show the results of torsion tests of five samples of heat-treated seamless steel tubing. A careful examination of these data will serve to show the importance of accurate torsion tests. The remarkable effect of the heat treatment of steel is very clearly shown.

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